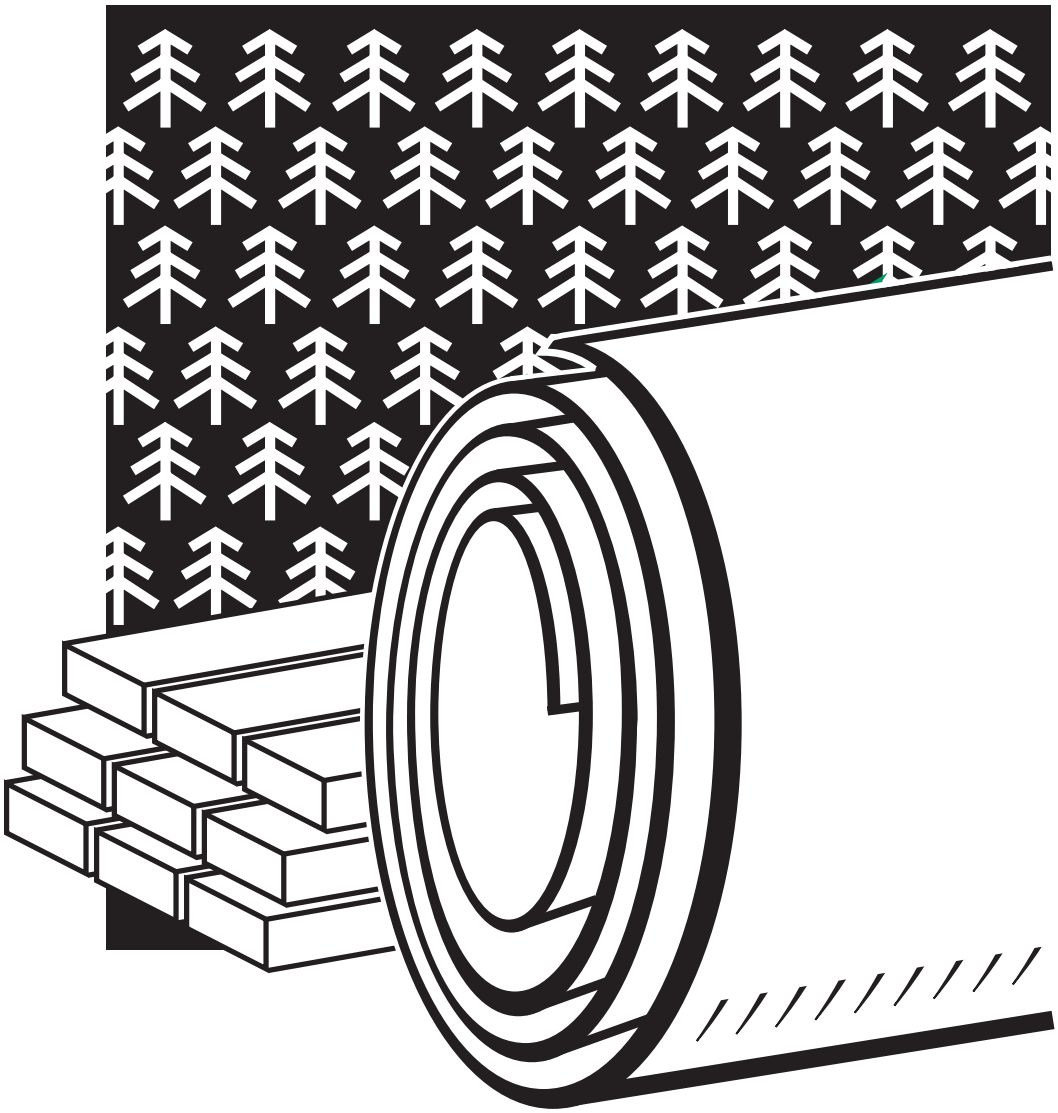
Forest Products Industry of the Future

Quarterly Status Reports

As of June 30, 2006



U.S. DEPARTMENT OF ENERGY

02-GA50113-09

Forest Products

Industry of the Future

Quarterly Status Reports

As of June 30, 2006

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Enhanced Raw Materials

Mill Processes: Pulping

Mill Processes: Papermaking

Mill Processes: Process Control

Mill Processes: Recovery / Environmental

Improved Fiber Recycling

Wood Processing

Dominant Negative Mutations of Floral Homeotic Genes for Genetic Engineering of Sterility in Forest Trees

Brunner: Oregon State University

ID13552

Project Title	**Development and Validation of Sterility Systems for Trees**
Covering Period	April 1, 2006 through June 30, 2006
Date of Report	July 31, 2006
Recipient	Oregon State University, Department of Forest Science, 350 Richardson Hall, Corvallis, OR 97331-5752
Award Number	DE-FC07-97ID13552
Contacts	Steve Strauss,: 541-737-6578,Steve.Strauss@oregonstate.edu Olga Shevchenko, 541-737-8482. olga.shevchenko@oregonstate.edu

Project Objective

The overall goal of this project is to develop and validate sterility systems in poplar that fulfill the basic requirements for commercial use. For this, sterility must be complete and stable over multiple growing seasons, cause no detrimental effects on vegetative growth, and successful transformation events must be identifiable via molecular tests when trees are still juvenile. Because of the inherent difficulties in achieving and demonstrating complete sterility in trees, our approach is to study alternate sterility systems that are mechanistically redundant in Arabidopsis and/or early-flowering tree systems. We will complete evaluation of dominant negative mutant (DNM) sterility constructs initiated under our previous grant and also test sterility constructs that are designed to engineer sterility via additional molecular mechanisms: floral cell ablation, overexpression of repressors of flowering, and high frequency gene-silencing. Constructs selected from lab and greenhouse evaluations, as well as constructs previously developed, will be tested in field trials of transgenic trees.

Background

We have isolated and characterized six poplar gene homologs of well-studied Arabidopsis genes that control the early stages of flower development (2). All but *LFY*/*PTLF* belong to a

Arabidopsis Gene	Function in Arabidopsis	Poplar Homolog(s)
AGAMOUS (AG)	Stamen & carpel identity	*PTAG1, PTAG2*
APETALA3 (AP3)	Petal & stamen identity	*PTD*
APETALA1 (AP1)	Flower initiation; sepal & petal identity	*PTAP1-1, PTAP1-2*
LEAFY (LFY)	Flower initiation	*PTLF*

family of transcription factors named after its highly conserved DNA binding and dimerization region, the MADS domain (3). We had planned to isolate a poplar homolog of the recently identified single-copy gene from Arabidopsis *NOZZEL (NZZ*), which is necessary for both female and male fertility. It differs from the genes listed above in that it acts at very late stages of flower development (4, 5). However, after intensive study we have concluded it is not a suitable candidate for near-term sterility systems due to its rapid evolution that makes isolation difficult, and very weak expression. Thus, we have stopped its development to ensure that the other goals of this proposal are met. Studies in Arabidopsis have identified moderately strong DNM transgenes, thus we are testing the same genes in poplar. Additional studies focus on the development of floral ablation and dsRNA-induced gene silencing (RNAi) sterility systems. Because overlapping gene function and the potential instability of transgene expression or

silencing make it difficult to be confident that sterility will be absolute throughout a tree's life span, redundant sterility constructs will be generated to overcome these difficulties.

Status

Because the project is nearly complete, we have focused on PCR confirmation and propagation of transgenic poplars to get enough transgenic events for establishment of a field trial to be planted in Summer–Fall of 2006. Currently we have produced and verified nearly 1,964 transgenic poplar events (Table1). Based on assumption to have 10 ramets per transgenic event we got propagated around 4,130 trees for 6K10 clone, 5,450 - for 717 clone and 5,830 plants for 353 clone. Transgenics with floral ablation transgenes and some DNM construct are the most difficult to produce. Due to strong non-specific floral expression, we were unable to produce any viable transgenic plants using the *PTAP1* ablation construct.

Table 1. Summary of confirmed independent transformation events with sterility or floral reporter control genes intended for field testing. Numbers of transgenic events planted in field in 2003 are shown in parentheses. IP- selection of transgenic lines in progress, NP- not planned, bold - enough transgenic lines for the field trial.

Construct type	No. of constructs	Construct	Clone 717	Clone 353	Clone 6K10
DNM	4		87	79	46
		AP1-M2	**21**	**13**	3
		AP1-M3	**27**	**31**	**25**
		AG-M2	**17**	**7**	1
		AG-M3	**22**	**28**	**17**
RNAi	20		367 (30)	388	421
		PTD-IR	**23**	**22**	**28**
		PTAG-IR	**24**	**24**	**25**
		PTAG-IR + MARs	NP	NP	**12**
		PTAP1-IR	**20**	**20**	**23**
		PTLF-IR	**25**	**21**	**22**
		PTAG-IR + PTLF-IR	**21**	**27**	**40**
		PTLF/PTAG-IR	**26**	**18**	16
		PTLF/PTAP1-IR	**22**	**22**	**41**
		PTAP1/PTAG-IR	**24**	**28**	**25**
		PTLF/PTAG/PTAP1-IR	**20**	**20**	**29**
		PAGL20-IR	**24**	**17**	**26**
		PAGL24-IR	**22**	**28**	**28**
		PFT-IR	**20**	**27**	**28**
		PFPFL2-IR	**21**	**29**	**24**
		PFPFL1-IR	**21**	**29**	NP
		PFT/PAGL20-IR	**13**	**17**	NP
		FT/AGL20-IR + FPFL1-IR	**11**	**12**	NP
		PMFT	**15(15)**	NP	**28**
		PCEN	**15(15)**	**27**	**26**

Overexpression	4		100 (38)	82	129
		PMFT	**19 (19)**	NP	**26**
		PCEN	**19 (19)**	**27**	**23**
		PSVP	**30**	**26**	**41**
		PAGL24	**32**	**29**	**39**
Ablation	5		65 (57)	12	13
		PTLF-BSB	**21(21)**	2	6
		PTLF-BOB	**17(17)**	2	NP
		PTLF-BNB	**19(19)**	0	NP
		PTAP1	0	0	0
		PTD	8	8	7
Reporter	3		62 (6)	74	39
		PTAP1::GUS + MAR	**24**	**25**	**13**
		PTAP1::GUS	**32**	**20**	**13**
		PTLF::GUS + MAR	**6 (6)**	**29**	**13**
Totals	**36**		**681**	**635**	**648**

The major planting that was planned for Spring of 2006 had to be postponed until fall due to delays in getting a contract for clearing of the site, leveling, and installation of an irrigation system bid and accepted. As a result of this delay, we had to repropagate a large number of plants to ensure they were not excessively pot-bound and ready for a much later planting than anticipated. In order to accommodate all of the constructs types and most possible number of events we had to clear around nine acres of land which was not under agriculture use. Currently land has been cleared, leveled, thoroughly disked and harrowed. We are presently in process of installing irrigation at this site.

Plans for Next Quarter

We will continue to do propagation of transgenics with DNM, RNAi floral ablation and other sterility constructs in preparation for establishment of a large field trial in summer-Fall 2006.

Milestones

ID Number	Task / Milestone Description	Planned Completion	Actual Completion	Comments
13552	Development and Validation of Sterility Systems for Trees Oregon State University, Strauss, PI	12/31/05		
Task 1	DNM sterility system evaluation in Arabidopsis	12/31/02	9/30/03	Four DNM transgenes selected for testing in poplar
Task 2	DNM sterility system evaluation in poplar and eucalypts	2/28/05		Evaluating select transgenes in poplar
Task 3	Evaluation of *PTAP1* promoter for floral ablation	11/30/04		Analysis of transgenic phenotypes in progress, but does not appear useful for ablation constructs due to cytotoxicity seen
Task 4	Isolation of poplar and eucalypts *NZZ*	4/1/03	10/30/03	Decided not a good candidate for sterility
Task 5	Evaluation of *NZZ* poplar promoter for floral ablation	3/30/05	12/30/05	Decided not a good candidate for sterility
Task 6	Evaluation of RNAi sterility systems	12/31/05	2/28/06	Picked up transgenic events with the lowest level of endogene expression
Task 7	Redundant sterility system testing	1/31/06		Ongoing, PCR confirmation and propagation of transgenic events
Task 8	Establishment of transgenic poplar field tests	9/31/06		Field-tests will extend beyond the grant period

Budget Data

Project Spending and Estimate of Future Spending							
Quarter	**From**	**To**	**Estimated Federal Share of Outlays***	**Actual Federal Share of Outlays**	**Estimated Recipient Share of Outlays***	**Actual Recipient Share of Outlays**	**Cumulative**
	Start*	6/30/04		615,947.32		195,296.91	811,244.23
3Q04	7/1/04	9/30/04	42,000	33,310.43	8,400	6356.06	39,666.49
4Q04	10/1/04	12/31/04	49,468	29,959.83	9,894	6,073.61	36,033.44
1Q05	1/1/05	3/31/05	35,000	31,775.38	7,000	6,328.94	38,104.32
2Q05	4/1/05	6/30/05	32,000	41,536.52	6,400	60,512.27	102,048.74
3Q05	7/31/05	9/30/05	28,000	47,318.18	5,600	25,563.92	72,982.10
4Q05	10/1/05	12/31/05	19,692	24,271.14	24,915	26,687.83	50.958.97
1Q06	1/1/06	3/31/06	5,000	12,770.38	867.17	9,166.19	21,936.57
2Q06	4/1/06	6/30/06	5,000	7,120.59	0	0	7,120.59
3Q06	7/31/06	9/30/06	2,190		0		
4Q06	10/1/06	12/31/06	2,000		0		
Totals				**844,009.77**		**336,085.73**	**1,180,095.50**

***Start is 9/01/97**

Performance and Value of CAD-Deficient Pine

Li: North Carolina State University

ID14436

Dr. Li was given permission to work on the final report and to bypass the quarterly for the period ending June 2006.

Engineering of Syringyl Lignin in Softwood Species Through Xylem-Specific Expression of Hardwood Syringyl Monolignol Pathway Genes

Joshi: Michigan Technological University, NREL

ID14440

QUARTERLY PROGRESS REPORT

Project Title: Improved Wood Properties Through Genetic Manipulation: Engineering of Syringyl Lignin in Softwood Species Through Xylem-Specific Expression of Hardwood Syringyl Monolignol Pathway Genes

Covering Period: April 1, 2006 to, June 30, 2006

Date of Report: July 14, 2006

Recipient: Michigan Technological University
1400 Townsend Drive, Houghton, MI 49931-1295
Congressional District: MI Ist

Award Number: DE-FC36-03ID14440

Subcontractors: North Carolina State University, Room 1 Leazer Hall, Campus Box 7514, Raleigh, NC 27695-7514
Matt Ronning, Associate Vice Chancellor
Ph: (919)-513-2148
11th Congressional District, NC.

Other Partners:

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Ph: (843) 851-4676, 1st Congressional District, SC.

(2) MeadWestvaco, P.O. Box 1950, 180 Westvaco Road, Summerville, SC 29484
David S. Canavera, Director of Tree Improvement
Ph: (843) 851-4774, 1st Congressional District, SC.

(3) Weyerhaeuser Company, WTC, 2D39, P.O. Box 9777, Tacoma, WA 98063-9777
Robert C. Eckert, Director of Strategic Technology
Ph: (253) 924-6503, 9th Congressional District, WA.

(4) International Paper Company, P.O. Box 7910, Loveland, OH 45140-7910
Richard B. Phillips, Senior Vice President-Technology
Ph: (513) 248-6001, 71st Congressional District, OH.

Contact(s): Chandrashekhar P. Joshi, Plant Biotechnology Research Center, School of Forest Resources and Environmental Science, Michigan Technological University, Houghton, MI 49931, 906-487-3480; cpjoshi@mtu.edu
(subcontract) Vincent L. Chiang, Forest Biotechnology Group, College of Natural Resources, North Carolina State University, 919-513-0098; vincent_chiang@ncsu.edu

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Project Objective:

Our long-term goal is to genetically engineer higher value raw materials with desirable wood properties to promote energy efficiency, international competitiveness, and environmental responsiveness of the U.S. forest products industry. The immediate goal of this project is to produce the first higher value softwood raw materials engineered with a wide range of syringyl lignin quantities.

Background:

The most important wood property affecting directly the levels of energy, chemical and bleaching requirements for kraft pulp production is lignin. Softwoods contain almost exclusively chemically resistant guaiacyl (G) lignin, whereas hardwoods have more reactive or easily degradable lignins of the guaiacyl (G)-syringyl (S) type. It is also well established that the reactive S lignin component is the key factor that permits much lower effective alkali and temperature, shorter pulping time and less bleaching stages for processing hardwoods than for softwoods. Furthermore, our pulping kinetic study explicitly demonstrated that every increase in one unit of the lignin S/G ratio would roughly double the rate of lignin removal. These are clear evidence that softwoods genetically engineered with S lignin are keys to revolutionizing the energy efficiency and enhancing the environmental performance of this industry.

Softwoods and hardwoods share the same genetic mechanisms for the biosynthesis of G lignin. However, in hardwoods, three additional genes branch out from the G-lignin pathway and become specifically engaged in regulating S lignin biosynthesis. In this proposed research we will simultaneously transfer aspen S-specific genes into a model softwood, black spruce, to engineer S lignin.

Status

During this quarter, the research continued on transformed callus selection, somatic embryo maturation and germination, transgenic seedlings regeneration, and greenhouse planting. The work is related to the planned Project Milestone, NCSU Tasks 3 (Transformation of black spruce with aspen *CAld5H*, *AldOMT* and *SAD* gene expression constructs via *Agrobacterium*-mediated multigene transfer and regeneration and propagation of transgenics) and Task4 (molecular genetic characterization of transgenic black spruce plants). Specifically, the work included: (1) subculture for continuous selection of the transformed calli using the constructs of aspen S-lignin genes under control of double 35S constitutive promoter (pBI-K35S-PtCAld5H, pBI-PtAldOMT, and pBI-PtSAD, designated as D35S construct set) and the constructs of the S-lignin genes under control of spruce xylem-specific promoter (pBI-PmP_{4Cl}-PtCAld5H, pBI-PmP_{4Cl}-PtAldOMT, and pBI-PmP_{4Cl}-PtSAD, designated as SXS construct set), (2) somatic embryo maturation and germination of the transformed calli, (3) regeneration of the transgenic seedling and greenhouse planting, and (4) molecular genetic characterization of transgenic seedlings.

Note: The late start of the project and prorated project budget have caused considerable interruptions to an effective, continual progress of the project. As a result, the timing and therefore progress on regeneration of transgenic plants, in particular, has been most seriously affected, requiring an extension of the project period. We are in the process of requesting a no-cost extension of the project to October 31, 2007.

1. Subculture for continuous selection

We have previously reported that we succeeded in the generation of about 20 black spruce embryonic callus lines from about 700 zygotic embryos. The screening of the callus lines yielded 2 lines, NBS01 and NBS02, suitable for somatic embryo regeneration and transformation. After the callus lines were selected, we proliferated the callus tissue to a large amount for transferring the D35S construct set and the SXS construct set. For NBS01 line, a total of 9 batches of transformation with the D35S construct set and 12 batches of transformation with the SXS construct set have been carried out. For NBS02 line, a total of 7 batches of transformation with the 35S construct set and 10 batches of transformation with the SXS construct set have been conducted. For each batch of the transformation, approximate 80 gram of the callus tissue was used and cultured in 10 petri dishes. After co-transformation, the calli were cultured in selection media for about 2-3 months. The selection of the transformed calli was through continuous subcultures with an interval of 2 weeks on the medium containing kanamycin antibiotics. After 2-3 month selection, usually, approximate 10% of callus masses were selected as transformed callus masses for the subsequent embryo maturation. Up to now, we have selected more than 150 masses transformed with each set of constructs.

2. Somatic embryo maturation and germination of the transformed calli

After 2-3 month selection culture, the positive callus mass was cultured onto embryo regeneration medium for embryo formation. About 8 -10 weeks culture is required to generate mature embryos. When somatic embryos were formed and grew to ~ 2 mm in length, they were harvested from the callus mass and cultured on the germination medium. Now the embryos are continuously harvested from various batches of the transformations. It usually took about 3 months for germination and elongation.

3. Regeneration of the transgenic seedling

The harvested embryos have been continuously germinated and grown to seedlings after cultured for about 4 month. We have tested and optimized various conditions for planting the transgenic seedlings in greenhouse. We have successfully planted 8 batches totaling more than 400 transgenic seedlings in greenhouse. The seedling survive rate is about 80%. We noticed the summer weather has affected the seedling growth.

4. Molecular genetic characterization of transgenic seedlings

From the greenhouse grown seedlings, 15 plants were analyzed with PCR determination for transgenics. The PCR results indicated that 7 plants were integrated with transgenes and 2 of them harbored 2 transgenes together.

Plans for Next Quarter

In next report period, we will continue to work on subculture and regeneration of the already-transformed callus tissues which now are at various culture stages. We will maintain the regenerated seedlings in a greenhouse. At the same time, we will continue to perform PCR confirmation and to screen promising transgenic plants for further characterization. Once the transgenic plants are screened, we will propagate them through an in vitro regeneration system we have already established.

Milestone Status Table:

ID Number	Task / Milestone Description	Planned Completion	Comments
NCSU Task 1	Cloning of spruce xylem-specific promoter	6/1/04	Complete
NCSU Task 2	Preparation of aspen *CAld5H*, *AldOMT* and *SAD* gene expression constructs and *Agrobacterium* strains	8/1/04	Complete
NCSU	Transformation of black spruce with aspen	6/1/06	

Task 3	*CAld5H*, *AldOMT* and *SAD* gene expression constructs via *Agrobacterium*-mediated multigene transfer and regeneration and propagation of transgenics		In progress & on schedule
NCSU Task 4	Molecular genetic and biochemical characterization of transgenic black spruce plants	6/1/06	In progress & on schedule
NCSU Task 5	Lignin content and S/G protocol establishment	6/1/05	Complete
NCSU Task 6	NIR-based characterization of cellulose and xylan contents	6/1/06	In progress & on schedule
NREL Task	Py-MBMS quantification of the S/G ratios and other lignin structural details for the finalist transgenic spruce	6/1/06	In progress
	Final Report	7/1/06	See "Note" in the "Status" section of this report

Patents: None

Publications/Presentations: None

Budget Data (as of date): The actual spending should reflect the money actually spent on the project in the corresponding periods.

Project Spending and Estimate of Future Spending							
Quarter	**From**	**To**	**Estimated Federal Share of Outlays***	**Actual Federal Share of Outlays**	**Estimated Recipient Share of Outlays***	**Actual Recipient Share of Outlays**	**Cumulative**
	Start	9/30/04		**600,406.09**		**128,491.96**	**728,898.05**
4Q04	10/1/04	12/31/04		**012,000.08**		**119,186.03**	**131,186.11**
1Q05	1/1/05	3/31/05		**000000.00**		**000000.00**	**0.00**
2Q05	4/1/05	6/30/05		**119,446.26**		**000000.00**	**119,446.26**
3Q05	7/1/05	9/30/05		**9,950.03**		**0.0000.00**	**9,950.03**
4Q05	10/1/05	12/31/05		**59,038.19**		**42,200.82**	**101,239.01**
1Q06	1/1/06	3/31/06		**71,182.66**		**0.00**	**71,182.66**
2Q06	4/1/06	6/30/06		**49,476.56**		**0.00**	**49,476.56**
3Q06	7/1/06	9/30/06	**499,339.32**		**160,309.14**		
4Q06	10/1/06	12/31/06					
1Q07	1/1/07	3/31/07					
2Q07	4/1/07	6/30/07					
3Q07	7/1/07	9/30/07					
4Q07	10/1/07	12/31/07					
Totals				**921,499.87**		**289,878.81**	**1,211,378.68**

* Update quarterly

Special note: A one-year no-cost extension for this project will be requested in September 2006.

General Note: DOE Laboratory partner spending should not be included in the above table. If a DOE Laboratory is a partner, report their spending and spend plan information in the table below (use separate tables if multiple DOE Laboratories are involved):
Note 1: Leave blank. Only the actual DOE/Cost Share amounts spent through 6/30/04 are needed.
Note 2: Amount for this quarter and subsequent quarters should be updated as necessary on a quarterly basis. Estimates need to be provided for the entire project. If spending for a given quarter is different than estimated, then the remaining quarter's estimates should be updated to account for the difference. Total DOE and Cost Share amounts should be the same as the Award amount.
Note 3: This should match the amount on the SF269A section 10.c. Column III (10.j. Column III on the SF269).
Note 4: This should match the amount on the SF269A section 10.c. Column II (10.j. Column II on the SF269).
Note 5: This should match the amount on the SF269A section 10.b. Column III (10.i. Column III on the SF269).
Note 6: This should match the amount on the SF269A section 10.b. Column II (10.i. Column II on the SF269).
Note 7: This should match the amount on the SF269A section 10.a. Column III (10.d. Column III on the SF269).
Note 8: This should match the amount on the SF269A section 10.a. Column II (10.d. Column II on the SF269).

DOE Laboratory Spending Table (if applicable):

DOE Laboratory Partner Spending and Estimate of Future Spending					
Quarter	**From**	**To**	**Estimated DOE Lab Amount***	**Actual DOE Lab Amount**	**Total**
	Start	6/30/04	Note 1		
3Q04	7/1/04	9/30/04	Note 2		
4Q04	10/1/04	12/31/04			
1Q05	1/1/05	3/31/05			
2Q05	4/1/05	6/30/05			
3Q05	7/31/05	9/30/05			
4Q05	10/1/05	12/31/05			
1Q06	1/1/06	3/31/06			
Etc.					
Totals					

Increasing Yield and Quality of Low-Temperature, Low-Alkali Kraft Cooks with Microwave Pretreatment

Compere: Oak Ridge National Laboratory

Agr id:10295

QUARTERLY PROGRESS REPORT

Project Title: Increasing Yield and Quality of Low-Temperature, Low-Alkali Kraft Cooks with Microwave Pretreatment

Covering Period: April 1, 2006 through June 31, 2006

Date of Report: July 31, 2006

Recipient: Oak Ridge National Laboratory (ORNL), operated by UT-Battelle
P. O. Box 2008
Oak Ridge TN 37831-6150

Award Number: DE-AC05-00OR22725

Subcontractors: H. Jameel, North Carolina State University (NCSU)
A. L. Fricke, Emeritus Professor, University of Florida

Other Partners: D. Parent, Communications and Power Industries

T. S. Bigelow, W. L. Griffith, and T. L. White, ORNL

Contact(s): A. L. Compere (865-574-4970) compereal@ornl.gov

Project Team: Drew Ronnenberg, DOE program manager; AF&PA program managers and mentors

Project Objective: The project goal is development of a predictive understanding of the effect of different microwave pretreatment parameters, including frequency, application pattern, and wood shape (logs or chips) and water content, on yield and quality of Kraft pulp produced using low-temperature or low-cooking-chemical digestion regimes. In addition to yield evaluation of Kraft pulps, pulp bleachability and handsheet quality (brightness, strength, freeness) will be evaluated for selected processing conditions. The ultimate project goal is development of proof-of-concept demonstration of a microwave/RF pretreatment process which could: 1) increase the yield of Kraft pulps, 2) decrease the cooking chemicals and cooking temperature required for production of a given quality pulp, 3) increase Tomlinson boiler throughput, or 4) decrease lime kiln energy usage.

Background: This project started six years ago as a two-year study supported by ORNL in-house funding to determine whether it was possible to develop “green chemistry” pulping technology. The project approach was to change the structure of wood to facilitate penetration of cooking chemicals. Pulping studies to evaluate the effect of pretreatment on ORNL

hardwood coppice were performed by Thomas Joyce of North Carolina State University through his small business, and Beloit and Bechtel staff provided advice on pulping technologies.

Initial proof-of-concept experiments were performed on small sections of wood (~200 g) in a small microwave unit, and later experiments evaluated pulp quality and yield of sycamore sections up to 3.75 inches in diameter treated with a high power microwave. The data consistently showed increased yield and lower Kappa number at conventional Kraft pulping conditions. Tom Joyce also found that a substantially smaller amount of cooking chemicals were required to achieve equivalent pulp quality. Bleaching and handsheet tests indicated that samples from pretreated wood were slightly brighter and slightly stronger than control samples. However, these proof-of-concept tests did not systematically evaluate processing schemes such as low-temperature or low-chemical cooks, which could minimize process energy and material requirements. Additionally, these tests did not systematically evaluate the effect of pretreatment on softwoods or address the ability to process chips. Both of the factors are critical to adoption of this process technology by the pulping industry.

The first part of this project as funded by the Industrial Technology Program had relatively modest goals: 1) to evaluate methods and develop technology for decreasing pulping chemicals, process time and energy (as H-factor), and cost required to produce a given amount of pulp and 2) to develop the data required to transfer the technology to both the forest products industry and manufacturers of large industrial microwave/RF systems. It was thought that shorter process times and lower cooking chemical use would permit an existing mill to produce more pulp at lower cost and with less environmental impact. However, initial expectations were a limited reduction, perhaps 2% active alkali (AA), in pulping chemicals and a modest reduction, perhaps 5%, in H-factor (time at temperature), a measure of process energy. It was also thought that the technology would facilitate pulping of oversized (12 mm thick) chips.

Using a small (100 mm diameter) continuous 915 MHz microwave applicator, the project team was able to show that very large sections (up to 100 mm long X 100 mm wide) of bark-on microwave pretreated hardwood harvested on the ORNL reservation could be pulped at greatly reduced cooking chemical concentrations (12% AA vs. 16-17% for untreated conventional chips) or significant reductions in H-factor (50%).

The project was extended and, as requested by advisors, the research team worked with industrial partners to develop a prototype industrial applicator which for use on chips, logs, and lumber. This was accomplished, and our industrial partner, Communications and Power Industries, constructed the applicator and subsidized its lease to ORNL. The applicator was specifically designed to provide information which will facilitate their scale up of this technology.

Using both softwood and hardwood chips, we have been able to show reductions in both process energy (45% reduction in H-factor for pine, 50% for sycamore) and in pulping chemicals (45% reduction in H-factor for pine, 40% for sycamore). Most important, reductions in cooking chemicals and in H-factor could be achieved in the same pulping runs. Additionally, screened

yield, handsheet properties, and kappa number equal or exceed those of paired, untreated, conventionally-pulped samples.

This year, reductions in pulping chemicals are becoming particularly significant because natural gas is typically used to fire kiln used to prepare lime used in white water preparation (chemical recycle).

To assess the potential impact on the industry, periodic energy and mass balance assessments have been prepared. Using sample cooks matched for kappa number and yield, but with varying pulping chemicals, the impact of lowered H-factor on mill throughput was also assessed. The results indicated that microwave pretreatment could, for the samples evaluated, increase batch mill throughput by 9% to 35% due to reduced sample pulping times.

Finding methods, like the hemicellulose separation reported below, which could increase the throughput of existing pulp mills, has the potential to facilitate adoption of microwave pretreatment technology. A mass, energy, and econometric evaluation shown below indicates the potential feasibility of this approach.

Status: During the last quarter, several different studies were conducted. Redisposition of single-step separated hemicellulose onto pulp was evaluated. Precipitation, separation, and use of black liquor hemicellulose has the potential to simultaneously increase both the yield of pulp from a mill and the solids concentration of black liquor fired to the boiler.

Initial studies indicated that up to 15% hardwood hemicellulose could be redeposited and retained in hardwood pulp provided that initial hemicellulose to pulp contact occurred at pH levels where the hemicellulose is soluble. Burst index of handsheets made from these samples increased with increasing hemicellulose content across the range. Tensile index reached a maximum at 10% hemicellulose content and remained at that level between 10 and 15%. Tear index reached a maximum at 2.5% hemicellulose, remained at that level for samples containing up to 10% hemicellulose, and decreased slightly above 10% hemicellulose.

It also appears possible to redeposit hardwood hemicellulose (xylan) on slightly-refined softwood linerboard pulps cooked to 99 kappa. Dissolved xylan was contacted with hydrated pulp and the slurry adjusted to pH levels between 7 and 9 and held at 30 to 70 C for two hours. Handsheets were prepared from this material. Untreated controls were prepared in the same fashion without addition of hemicellulose.

The highest increases in hemicellulose deposition (15%) and tensile, tear, and burst strength were found at the highest pH (9) and the lowest contact temperature (30 C). Addition of polydiallyldimethylammonium chloride (poly-DADMAC) to hardwood pulp handsheets was also evaluated. Although addition of poly-DADMAC did not significantly increase absorption of hemicellulose, it did provide a consistent, if lower, increase in tensile, burst, and tear strength across the pH range.

A portable continuous wave microwave system capable of treating enough chips for small digester studies is being evaluated. This portable system would permit the project team to perform a short series of studies to evaluate pulping immediately after pretreatment and chip soaking. To be sure that the pulping performance of chips pretreated using this system was comparable to that of chips pretreated using other applicators, white oak chips sample were pretreated, soaked in pulping liquor, drained, and sent to NCSU for pulping evaluation. In the last quarter, white oak, the most difficult hardwood to pretreat, was assessed. Decreases in H-factor and chemicals required to pulp white oak were comparable to those obtained with two different types of microwave applicators.

This quarter, mill run chips of Southern pine, a typical Southern softwood, were assessed. As shown in Table 1, the controls 1 and 2 were done on one digester and 3 and 4 were done another digester. All the controls gave similar results. The yield from the controls was slightly below that expected, possibly due to high juvenile wood content. In the control, at an alkali charge of 19%, the kappa number was about 30.

With the microwaved chips, the kappa number was 20 at similar pulping conditions. With microwaved chips the kappa number was lower than the control at an alkali charge of 15% active alkali (AA). At the same alkali charge, the H Factor could also be decreased from 1900 to 1150. The portable batch microwave gave results comparable those obtained with continuous systems.

Table 1. Pulping results from new microwave with southern pine

Cook #	AA, %	Sulfidity, %	H-Factor	Screened Yield, %	Rejects, %	Total Yield, %	Kappa
Control #1	19	25	1900	39.2	0.15	39.4	32.6
Control #2	19	25	1900	38.5	0.31	38.8	32.4
Control #3	19	25	1900	37.6	0.12	37.7	30.2
Control #4	19	25	1900	38.4	0.27	38.7	29.6
Treated #1	19	25	1900	38.7	0.02	38.7	20.2
Treated #2	15	25	1900	40.1	0.01	40.1	25.2
Treated #3	19	25	1400	40.5	0.04	40.5	22.6
Treated #4	19	25	800	42.9	0.1	43.0	41.5
Treated #5	19	25	1150	42.3	0.05	42.4	28.0

Plans for Next Quarter:

Evaluations of the portable applicator on softwood chips will be performed and arrangements will be made to take it to NCSU so that pretreated chips can be pulped on a schedule comparable to that of a mill. A series of pulp cooks at progressively increasing sizes are planned to improve understanding of process scale up. These will include hardwood and softwood Kraft cooks to evaluate minim for H-factor (energy) and pulping chemicals. The

softwood and hardwood soda and soda – anthraquinone cooks which incorporate anthraquinone with the pulping liquor used to soak chips will be completed. Studies to evaluate the effect of hemicellulose separation on pulp mill mass and energy will continue.

Patents: None.

Publications/Presentations: None.

Milestone Status Table:

ID Number	Task / Milestone Description	Planned Completion	Actual Completion	Comments
1.1	Complete preliminary small digester experiments evaluating low-temperature and low-cooking-chemicals pulping	10/31/2001	11/02/2001	Presented as TAPPI Pulping Conference paper
1.2	Complete draft preconceptual design report	10/31/2001	09/30/2001	
1.3	Initiate softwood studies	09/30/2002	09/30/2002	
1.4	Larger cooks and papermaking evaluations	09/30/2002	09/30/2002	
2.1	Pretreatment tests of logs	03/31/2003	03/31/2003	
2.2	Complete initial handsheet and bleaching tests	09/30/2003	09/30/2003	
2.3	Initiate large cooks to prepare enough pulp for evaluation	12/31/2003	03/15/2004	Funding received late, equipment problems.
2.4	Handsheet and bleaching tests	09/30/2004	09/30/2004	
2.5	Softwood and hardwood cooks with larger test facility (TBD)	2005	2006	Funding limited, equipment unavailable during move

Budget Data (as of July 25):

Phase / Budget Period			Approved Spending Plan			Actual Spent to Date		
	From	To	DOE Amount	Cost Share	Total	DOE Amount	Cost Share	Total
Year 1	10/99	9/00	200000	108000	308000	68391	108000	176391
Year 2	10/00	9/01	350000	108000	458000	251715	108000	359715
Year 3[a]	10/01	9/02	350000	108000	458000	279120	108000	387120
Year 4[a]	10/02	9/03	372000	122200	494200	238722	110200	348922
Year 5	10/03	9/04	395000	134200	529200	308692	106200	414892
Year 6[b]	10/04	9/05	421000	104200	525200	212325	110200	322525
Year 7	10/05	9/06				190255	55100	245355
Totals			2088000	684600	2772600	1549220	705700	2254920

[a]Only $250,000 in DOE funding was received during FY.
[b]Only $120,000 in DOE funding was received during FY.

Hemicellulose Extraction and Its Integration in Pulp Production

Van Heiningen: University of Maine

GO14306

QUARTERLY PROGRESS REPORT

Project Title: **Integrated Forest Products Refinery (IFPR)**

Covering Period: April 1st, 2006 – June 30st, 2006

Date of Report: August 5th, 2006

Recipient: **University of Maine, Corbett Hall, Orono, ME, 04469**

Award Number: FC36-04GO14306

Other Partners: International Paper

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Project Objectives:

Hemicelluloses will be extracted from residual wood chips prior to pulping. The extract is then recontacted with kraft fibers to increase pulp yield and quality. The remaining hemicelluloses in the extract constitute a feedstock for sugar based polymers and chemicals. The sugar-based polymers may replace fossil fuel based chemicals and resins in wood composites for the development of bio-composite materials.

The objectives of the project are organized in the following tasks:
1. Extraction of Hemicelluloses from Residual Wood Chips and its Integration in Pulp Production.
2. Development of Bio-Composite Materials using cellulosic fibers and polyesters.

The organizations which will work on the above listed 2 tasks are: The University of Maine and International Paper. Specifically, the University of Maine will address the laboratory experiments for tasks 1 and 2, while International Paper will take the lead on the industrial implementation of the findings of Task 1. The tasks will be executed over a period of 3 years.

In task 1 the benefits of hemicellulose extraction will be quantified in terms of: improved pulp yield and quality, decreased overall alkali consumption during kraft cooking, and a reduced organics load going to black liquor recovery. Extraction, pulping, oxygen delignification and pulp bleaching trials will be performed at the Pulp and Paper Process Development Center of the University of Maine to determine the optimum process conditions for the several operations involved in this task.

Task 2 concentrates on the production of Sheet Molding Compounds (SMCs) using natural fibers (such as micro-crystalline cellulose, wood pulps, and agricultural fibers rather than the commonly used glass fibers) and conventional polyester resin systems. The manufacturing operating parameters and properties of the SMCs will be determined, and the technical, process and marketing issues associated with the production of SMCs will be identified.

Background:

Managed forests in the United States have enormous untapped potential as sources of bioenergy and biomaterials. Realizing this potential requires an approach that integrates new separation and conversion technologies with existing forest products procurement, production and delivery infrastructure.

US chemical pulp mills receive more than 120 million dry tons of wood each year. The objective of this project is to evolve existing chemical pulp and wood product mills into Integrated Forest Products Refineries (IFPRs) that produce new biomaterials and export renewable energy while continuing to meet growing demand for pulp and paper products. The transition to IFPRs will be enabled by economic returns from improved ongoing pulp and paper production, while producing additional biomaterials and renewable energy.

In an IFPR it is most cost effective to use trees principally for solid wood production, while the residual wood fiber and biomass (bark) are used for pulp and power production respectively. The dominant alkaline pulping process is then used to release the highest quality fiber for pulp production from essentially all hardwood and softwood species with

minimal environmental impact. The pulp yield is only about 50% because most of the hemicelluloses and almost all the lignin end up in the spent pulping stream, called black liquor. The black liquor is combusted for steam and electricity generation, while the dissolved inorganic cooking chemicals are recovered and recycled for pulping. Since the heating value of the carbohydrates is only half that of lignin, the combustion of dissolved hemicelluloses does not constitute optimal economical use of this resource. Therefore, in the present IFPR a significant amount of the hemicelluloses are extracted from the residual wood chips prior to pulping. The relatively pure extract of hemicellulose oligomers is then recontacted with the delignified fibers to increase pulp yield and quality. The remaining hemicelluloses in the extract may be used as a feedstock for the production of ethanol, sugar based polyester polymers or other chemicals. The waste streams of the conversion processes will be combined with the black liquor to recover the heat value, and recycle sodium and/or sulfur. The sugar-based polymers may serve to replace fossil fuel based chemicals and resins in wood composites for the development of bio-composite materials.

The economic, energy and environmental benefits of the project are estimated assuming a pulp yield increase of 2% compared to conventional alkaline pulping, and implementation of the hemicellulose extraction technology in 50% of the chemical pulp mills. The higher pulp yield leads to an extra pulp production of 1.2 million tons at a value of about $600 million. The incremental pulp production is equivalent to an additional income of about $30/ton pulp produced. For the same scenario it is envisioned that the mills will produce 2 million tons of sugar-based chemicals and resins, representing an additional revenue stream of $2400 million/year or $80/ton pulp produced. The production of hemicellulose-based polyesters could save up to $85x10^{12}$ BTU/yr in energy in the year 2020 and $218x10^{12}$ BTU/yr when fully implemented.

Summary of Previous Work Completed on Task 1:

Extraction/Kraft Cooking of Southern Mixed Hardwoods

Southern mixed hardwood chips obtained from International Paper were extracted with pure water and aqueous alkaline solutions. The extracted chips were then cooked at standard conditions (160 °C, 12-15% EA charge, L/W of 4.5 L/kg and variable H-factor). Variables were the temperature of extraction, the extraction time, the amount of alkali charged and the form of the alkaline species. It was found that about 10% of the organics may be extracted from the wood chips with pure water or 10% NaOH. Unfortunately the total yield of the final kraft pulp after water extraction is lower than that of the control (i.e. no extraction), and the yield loss increases with extraction temperature. For example at 150 °C the total pulp yield is more than 5% lower than the control, and at 160 °C the yield loss is almost 7 %. Extraction with 10 % NaOH also gives a lower pulp yield, similar to that obtained with pure water extraction. For pure water extraction the yield loss after cooking is mostly due to xylan loss, while the yield loss after alkaline extraction and cooking is due both to cellulose and xylan loss.

To avoid the large (5-7%) pulp yield loss experienced with water and caustic extraction, we have adopted a different approach. With this new approach we are now able to increase the pulp yield (based on the original od weight of wood) to a level essentially equal or even 1% higher than that of a kraft cook control. Other benefits from this approach are:

- A reduction in EA charge during cooking by 3%
- An increase in delignification rate of up to 40%
- A reduction in the amount of rejects at higher kappa numbers
- A decrease in organic load to the recovery boiler by up to 8% based on od wood

An invention disclosure has been written up, and patents are being filed by the University of Maine and International Paper.

Gel Permeation Chromatography (GPC) of Extracts

GPC analysis of the extracts was initially preformed using water with the addition of 0.1M $NaNO_3$ as eluent. However, the limited solubility of the hemicelluloses as well as their tendency to form aggregates in water has lead us to change the analysis protocol. In the new method we have replaced the aqueous 0.1M $NaNO_3$ eluent by a DMSO:water (90:10) mixture containing 0.05M LiBr. GRAM columns from polymer standard service (PSS) specifically developed to handle organic-aqueous mobile phases are used in series. The detectors of our system are a UV detector (Viscotek VE 3210), a RALLS-VI detector (Viscotek 270 dual detector), and a RI detector (Viscotek VE 3580).

With the new GPC analysis using DMSO/H2O (90:10) as the mobile phase hemicellulose concentrations were measured in the extract ranging from 2 to 8 mg/g extract, and the DP of the dissolved hemicellulose polymers ranges from 130 to 250. The dissolved lignin content was measured separately using UV and a commercial lignin (Indulin AT) for calibration. The dissolved lignin content generally ranges from 2 to 5 mg/g extract. The sum of the mass of the dissolved hemicelluloses, lignin and inorganics (determined from the weight of the ash after air burning the freeze-dried solids at 550 °C), generally represents about 80 % of the centrifuged dissolved solids in the aqueous extract. The non-dissolved solids in the DMSO/H2O represent about 20 % of the total weight of the centrifuged dissolved solids in the aqueous extract, thus bringing the mass balance close to 100%.

Readsorption of Extract on Kraft Pulp

Adsorption experiments with the novel extract were conducted to study the hemicellulose adsorption on pulp. The extract was mixed in a plastic bag with the unbleached kraft pulp obtained after extraction and kraft cooking. After vacuum filtration, washing and drying, the adsorption yield and kappa number was measured. The results indicate that a yield increase of almost 2% can be achieved. The kappa number after adsorption was about 2 to 3 units higher than that of the brownstock pulp.

Bleaching of the Extracted and Non-Extracted Hardwood Pulps

Two hardwood pulps obtained after the novel extraction method (Kappa number of about 18 and 28% ISO brightness) performed on the mixed southern hardwood chips were bleached using a standard $D_0E_PD_1$ sequence. The ISO brightness obtained for two different chlorine dioxide (and NaOH) charges were 81.5 and 87%, exactly the same as for the control kraft pulp (also Kappa number of about 18 and 28% ISO brightness). This shows that the pre-extraction does not influence the bleaching response of the pulps.

Extraction/Kraft Cooking of Loblolly Pine

Loblolly Pine chips were extracted with pure water and different aqueous solutions containing sodium and sulfur salts. The following conclusions may be drawn for Loblolly Pine extraction with pure water:

1. The wood weight loss ranged from 10.7% at the mildest extraction condition of 15 minute at 170^{o}C to 24% at the most severe condition of 90 minutes at 190^{o}C.

2. The wood weight loss yield as well as dissolved total sugar yield, total hemicellulosic sugar yield, polymeric sugar yield, cellulose yield and lignin yield are all satisfactorily described by the H-factor variable, which combines the effect of time and temperature during extraction. A single curve is obtained for each yield at the three different temperatures.

3. The mono-sugar yield increases with H-factor up to a maximum of almost 5% on od wood basis at an H-factor of about 2500 hours, and then decreases. This behavior is consistent with that of a consecutive reaction where the monosugars are first formed by hydrolysis from the polymeric sugars, and then the monosugars are further degraded by hydrolysis to furfural, hydroxymethylfurfural, levulinic acid and other degradation products.

4. A maximum polymeric sugar yield of about 8 % (on od original wood) in the extract is obtained at an H-factor of about 500. The lignin content at this H-factor is about 0.5%. This level of extraction may be obtained by extraction for about 60 or 25 minutes at 160 and 170 ^{o}C respectively.

Summary of Previous Work Completed on Task 2:

Production and Testing of Sheet Molding Compounds (SMCs)

A compression mold made of carbon steel was designed and fabricated to produce a Sheet Molding Compound (SMC) of approximately 18 cm by 18 cm. To make the molded SMC, an industrial B-stage SMC was acquired from AOC Resins. The B-stage SMC was successfully compression molded into C-stage SMC at the Advanced Engineered Wood Composites Center,(AEWC). Samples of 7mm x 3 mm x 45mm were cut from the SMC. These samples were dried to constant weight, then sanded to 1.8 mm

thickness for DMTA analysis from -60ºC to 260°C at 1 Hz frequency and 1°C per minute ramp rate with a MKIV-Rheometrics DMTA to obtain physical property and environment aging data for the SMC. The Tan Delta for the hydro thermally soaked samples was determined with the DMTA and was used to make a hypothesis about the possible chemical reactions taking place in the SMC matrix. The hydrothermal tests simulate an accelerated exposure of the SMC to the environment.

Initial results have indicated that the instrument noise was too high for good reproducibility. This was addressed by moving the instrument to a vibration minimized area and placing the instrument on a vibration damping stone. This isolation has removed environmental noise allowing improved data collection.

Hydrothermal aging of the SMC has been completed with DMTA testing. The results agree with previous literature results in many aspects. The peaks indicating a thickening reaction for the polyester and metal oxide intensified with hydrothermal aging. The peak intensity for the glass transition of poly(vinyl acetate) decreased with aging as has been previously reported. However, HPLC results indicate that no acetic acid was formed during the hydrothermal aging. This disagrees with literature reports of the hydrothermal decomposition of poly(vinyl acetate) degrading to poly(vinyl alcohol) and acetate. The HPLC indicated the presence of large molecules in the aging solution. Considering the decrease in intensity for the poly(vinyl acetate), the large molecules may be some of the low profile additive. Since no glass transition peak was found for poly(vinyl alcohol) in previous work, and in the current investigation, the hypothesis that the large molecules in the aging solution may be poly(vinyl acetate) is plausible. Styrene was run using HPLC and compared with the aging solution and is shown to be leaching into water.

Inverse gas chromatography, (IGC), with kenaf fibers has been done. Initial experimental work has focused on getting meaningful results by proper packing of IGC columns and designing experiments. Experiments were conducted at three temperatures, 25°C, 35°C, 45°C. Inverse gas chromatography is done to characterize fiber and matrix surface energies to determine appropriate fiber sizing agents.

Prepreg SMC was donated by AOC resins; kenaf by Kenaf industries, glass from Owens Corning, and peroxide initiator from Akzo Nobel. This material will be used to compound SMC with glass and natural fibers.

Project Status Task 1

Extraction/Kraft Cooking of Loblolly Pine

The objective of this work is to establish the extraction operating conditions for Loblolly Pine chips which lead to selective hemicellulose removal, without causing significant damage to the cellulose remaining in the wood chips. During the first quarter of year 2 the extraction operating variables were investigated. During 2nd quarter the digester system was rebuilt and recalibrated following a incident which left the equipment seriously damaged. New operational procedures were established to prevent the occurrence of such an incident in the future. Also the temperature recalibration with a calibrated RTD set-up showed that previously the actually digester temperature was about 8 °C too high at a setpoint of 170 °C. During this quarter we performed cooks at 170 °C, 15% EA and 30% sulfidity on Loblolly Pine chips previously extracted with pure water at an intensity of 100, 200 and 500 H-factor. The results are compared with control cooks performed on the fresh chips (no pre-extraction) at the same conditions as those on the extracted chips as well as a higher EA charge of 17%.

The extraction yield loss determined due to water extraction at the three H-factor intensities are listed in Table 1. It shows that the amount of material extracted increases from 2 to 8 % on od wood when the extraction intensity is increased from an H-factor of 100 to 500 hours. From our previous work it is known that at 500 H-factor a significant amount of the dissolved wood is present in the extract as oligomeric hemicellulose

Table 1. Yield loss during extraction and pulping for pre-extracted and fresh chips

	Control cook at 15% EA	Extraction at 100 H-factor	Extraction at 200 H-factor	Extraction at 500 H-factor
Extraction yield loss (% on od wood)	0	2	5	8
pH of extract	--	4.4	4.2	4.1
Total pulp yield at 30 Kappa (% on od wood)	45	45	42	39
Pulping yield loss at 30 Kappa (% on od wood)	55	53	53	53

The development of the kappa number versus cooking time for the three extraction conditions and two control cooking conditions are shown in Figure 1. It shows that the kappa numbers at the same cooking time are lower for the extracted chips compared to the control cooks at 15 %EA.

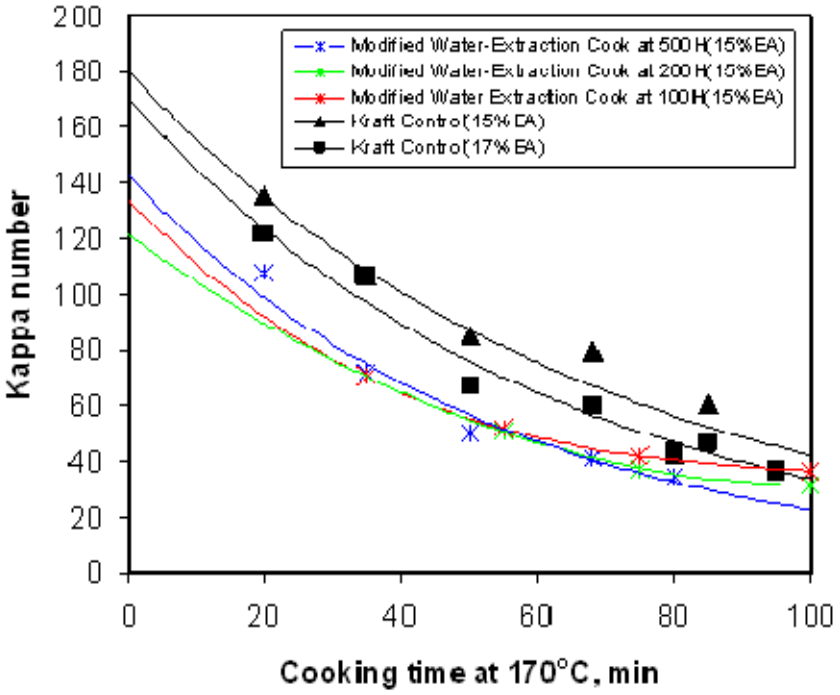


Figure 1. Kappa versus cooking time for water extracted and control chips

The kappa number versus cooking H-factor is shown in Figure 2. The results show that an H-factor of about 1400 hrs is needed to obtain a kappa number of about 30 for the 500 H extracted chips, while the control cook at 15% EA requires a significantly higher H-factor. Even if the EA charge of the control cook is increased to 17 % the H-factor required to produce a 30 kappa pulp is still about 1500 hours.

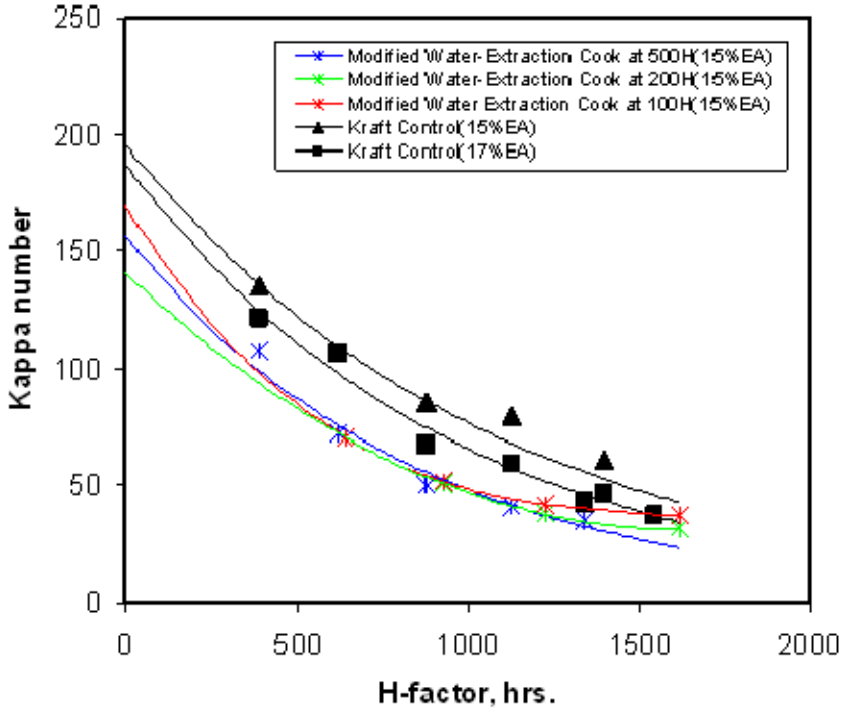


Figure 2. Kappa number versus H-factor for water extracted and control chips

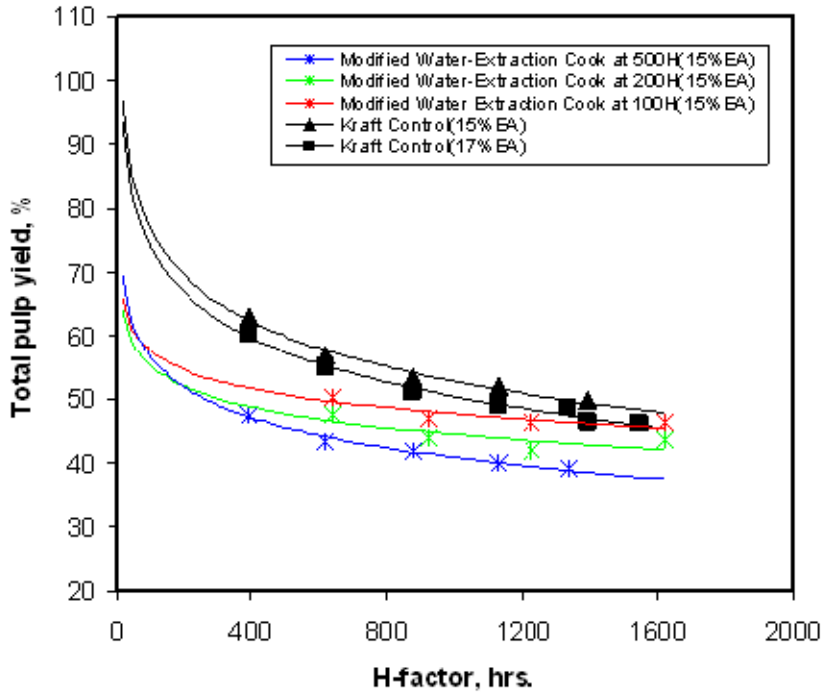


Figure 3. Total pulp yield (on od wood) versus H-factor for water extracted and control chips

The total pulp yield based on the original oven dried fresh chips as a function of H-factor is shown in Figure 3. It shows that the total pulp yield (on od wood) of the extracted

chips (also accounts for extraction weight loss) is lower than that of the control cooks especially at short cooking times.

The total pulp yield (on od wood) versus kappa number is shown in Figure 4. It can be seen that the total pulp yield for the pre-extraction cooks is lower than the control cooks, except for pre-extraction at an H-factor of 100 hours where the total yield is the same when cooking until a low kappa number of 30. The total pulp yield at 30 kappa number of the three pre-extraction cooks and the reference cook are also listed in Table 1. It can be seen that the pulp yield is 3 and 6% lower than the control with pre-extraction at H-factors of 200 and 500 hours respectively. This implies that after pre-extraction about 53% of the wood mass is dissolved during cooking until a kappa number of 30 compared to 55% for the reference pulp of 30 kappa. The only slightly smaller removal of wood mass during pulping after pre-extraction suggests that the carbohydrate removal process is mostly unchanged. Since the principal carbohydrate removal process for softwoods during pulping is alkaline peeling which consumes a considerable amount of the charged alkali, it is expected that the alkali consumption during pulping is also not much affected by pre-extraction. This can be seen in Figure 5 showing the residual effective alkali (REA) remaining after pulping to different kappa numbers for the pre-extracted and control cooks.

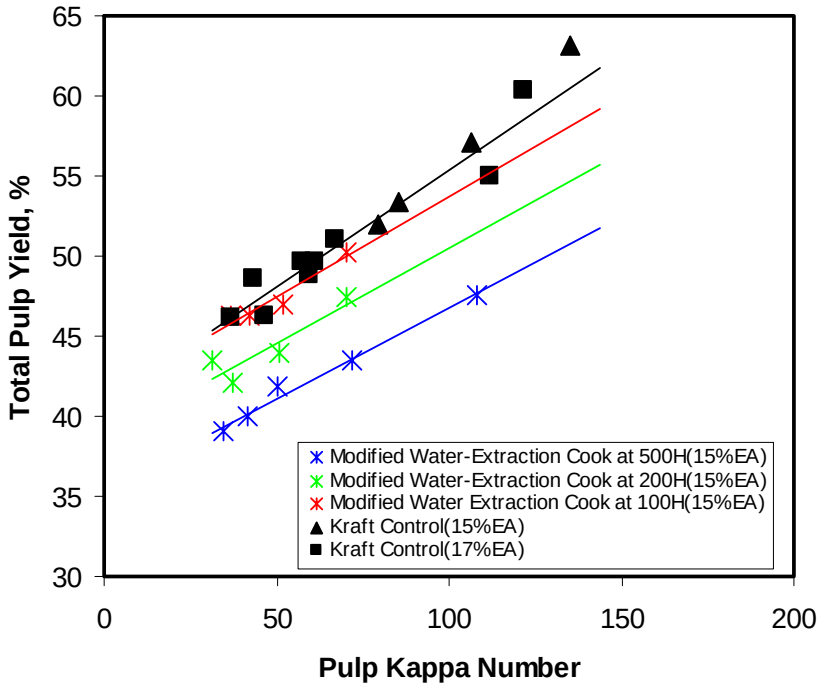


Figure 4. Total pulp yield (on od wood) versus kappa number for water extracted and control chips

Conclusion

Up to 8% of the wood may be pre-extracted from Loblolly Pine when the pre-extraction intensity is increased to an H-factor of 500 hours. Subsequent pulping to a kappa number of 30 leads to significant reduction in required cooking H-factor, but also to a pulp yield which is 3 and 6% lower than the control at pre-extracting H-factors of 200 and 500 hours respectively.

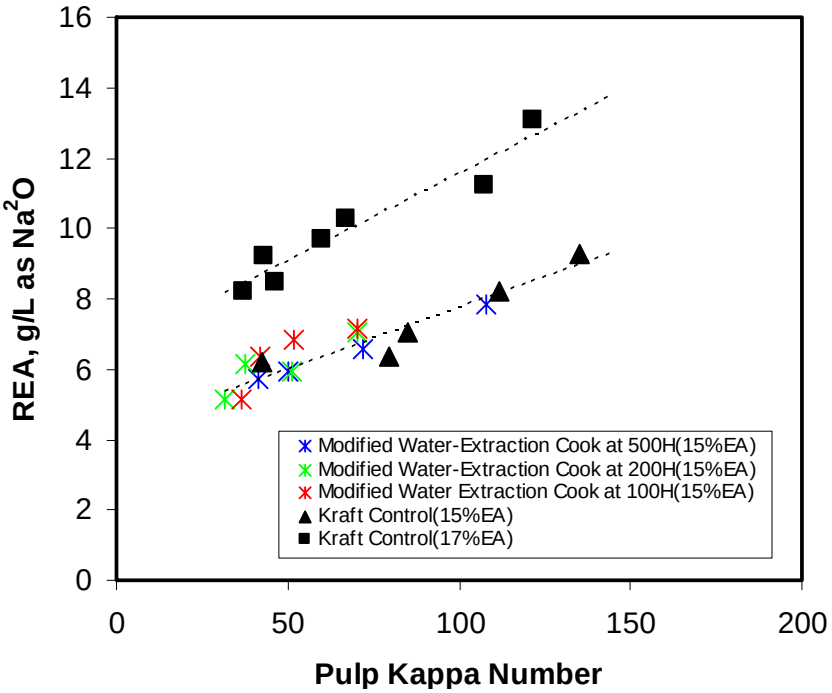


Figure 5. Total pulp yield (on od wood) versus kappa number for water extracted and control chips

Project Status Task 2

Production and Testing of Sheet Molding Compounds (SMCs)

HPLC was conducted to try to determine the presence of acetic acid or other chemicals in the aging solution. Figure 6 shows the HPLC graph of the aging solution.

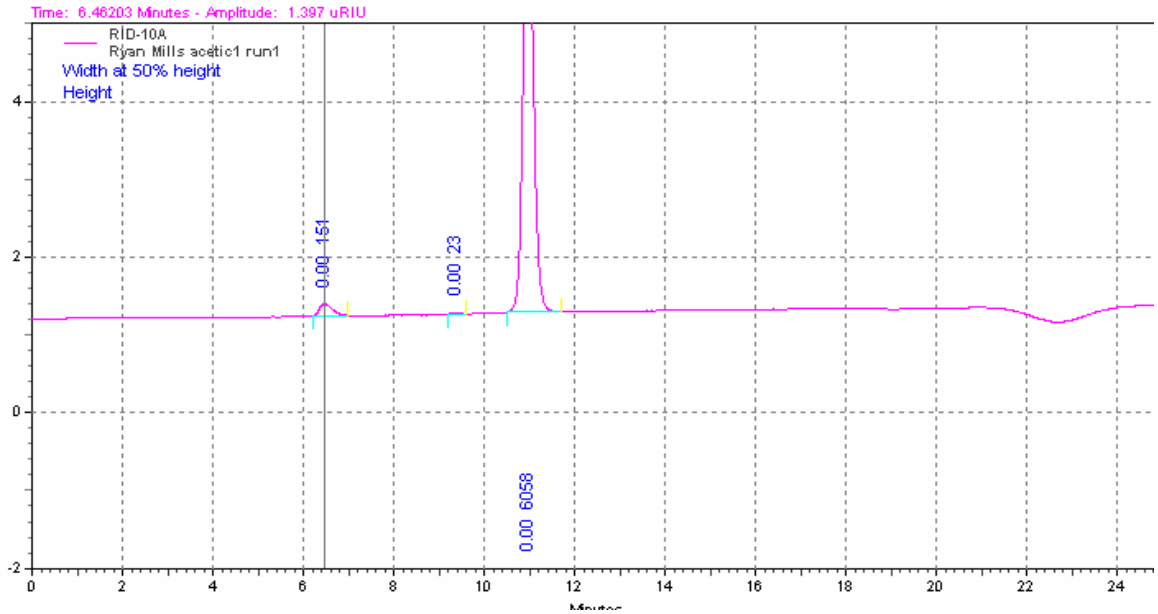
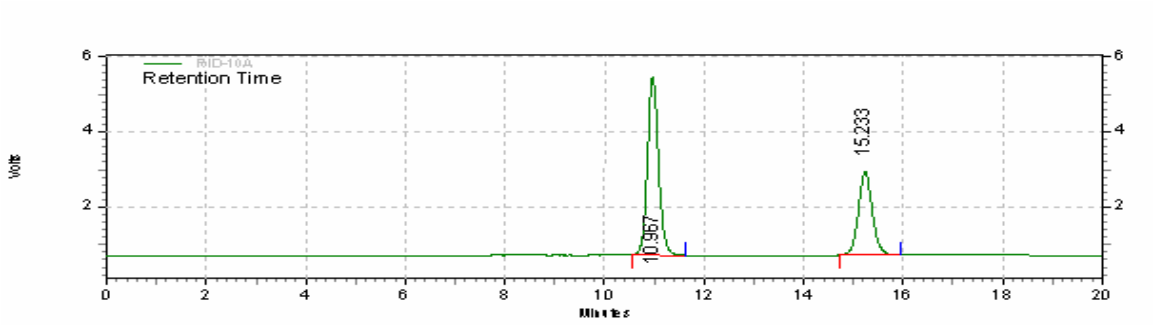


Figure 6. HPLC scan of aging solution for SMC

In the scan three peaks are seen at 6.5 minutes, 10.8 minutes, and an inverted peak at 22.5 minutes. If poly(vinyl acetate) is converted to poly(vinyl alcohol), acetic acid should be produced. An acetic acid sample was run with fucose as a standard reference (see Figure 7).



RID-10A Results

Retention Time	Area	Area %	Height	Height %
10.967	72656	62.95	4750	68.16
15.233	42769	37.05	2219	31.84
Totals	115425	100.00	6969	100.00

Figure 7. HPLC of acetic acid and fucose

Two peaks are seen with fucose at 10.9 minutes and acetic acid at 15.2 minutes. The acetic acid peak at 15.2 minutes doesn't correspond to any of the peaks in the aging solution indicating that acetic acid is not present in the aging solution. Based upon the composition, styrene was hypothesized to be one of the chemicals leaching from the SMC during aging. Therefore, another HPLC run with styrene using the same column was performed (see Figure 8).

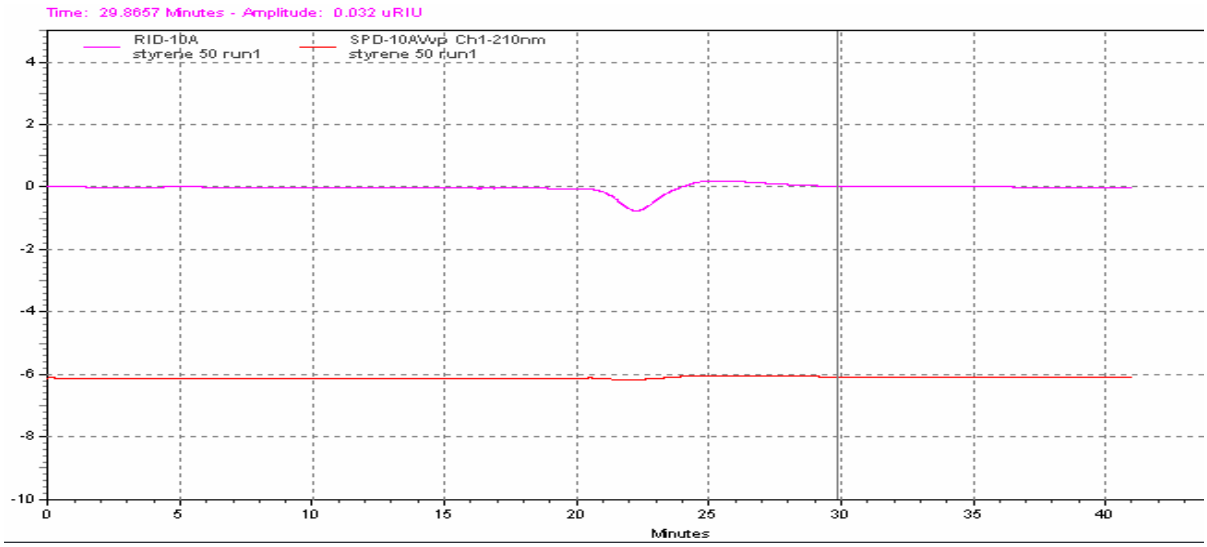


Figure 8. HPLC of styrene

In figure 8 an inverted peak at 22.5 minutes is seen for the styrene. Thus styrene is one of the molecules being leached out of the SMC. The other two peaks are still unidentified but are thought to be PVAc and residual or unreacted polyester.

IGC testing has been performed on kenaf fibers. Repeatability of experiments, the selection of proper acid-base probes for determining the character of the polyester interactions with the fibers, and probes based upon structural interactions have been completed. Continued runs with kenaf will be performed to optimize the process; cellulose and glass fibers will be run also. The sizing and B-Stage SMC fabrication will be based upon the surface energy analysis from IGC testing.

Plans for Next Quarter
Task 1:
Extraction/Kraft Cooking/Hemicellulose Adsorption/Bleaching of Loblolly Pine

1. Explore new pre-extraction methods of Loblolly Pine chips with the objective to minimize the pulp yield loss during subsequent kraft cooking compared to that of fresh unextracted chips.

2. Perform adsorption experiments with hemicelluloses extracted from Loblolly Pine chips on corresponding kraft pulp fibers to obtain a higher yield.

3. Determine the bleaching response of the pre-extracted Loblolly Pine kraft pulps.

Task 2:
Production and Testing of Sheet Molding Compounds (SMCs)

1) Fabricate SMC sheets at AEWC at University of Maine made with varying degrees of biobased reinforcement for testing and comparison with the standard "industrial" synthetic SMC. All material has been acquired and the B-Stage SMC is being fabricated.

2) Further HPLC analysis will be performed to identify the chemicals being leached from the SMC. This information will be useful for understanding the weather ability of the material. The weather ability of the material is important, as industrial standards require this material to perform in service for a minimum of 10 years.

3) The biobased fibers are being analyzed using inverse gas chromatography (IGC) to determine surface energies and to determine appropriate sizing agents for the fibers. This will also be done with the polyester prepreg material received from AOC resins. Following IGC analysis, sizing agents will be purchased and B-stage SMC will be fabricated with varying degrees of reinforcement, with and without sizing, and compression molded After compression molding of the B-stage SMC, tan delta and storage modulus curves will be determined with DMTA, impact testing ASTM D 256-00, tensile testing ASTM D 638-01, and water uptake as has been done previously.

Presentations by Adriaan van Heiningen

"Integrated Forest Products Refinery (IFPR)"
ITP Peer Review Meeting Atlanta, GA, April 5th and 6th, 2006

"**Forest Biorefinery Research at the University of Maine"**
NRBP (Northeastern Regional Biomass Program) Steering Committee Meeting, Latham, NY, May 25th, 2006

"**Project Review Meeting; Integration of Extraction of Hemicelluloses in Pulp Production"**
International Paper Corp., Loveland, OH, June 20th, 2006
(note: for confidentiality reasons this presentation is not attached)

Milestone Status Table:

ID Number	Task / Milestone Description	Planned Completion	Actual Completion	Comments
1.1a	Explore optimal extraction conditions for hardwood using ASE 100 of Dionex	3/31/05	10/31/05	
1.2a	Complete extraction and cooking of hardwood chips with profiling digester	8/31/05	7/31/05	
1.3a	Complete the deposition study of hardwood hemicelluloses on kraft pulp	8/31/05	8/31/05	
1.4a	Complete oxygen delignification of hard wood kraft pulp with adsorbed hemis	8/31/05	8/31/06	
2.1a	Produce SMCs from conventional polyesters	12/31/04	12/31/04	
2.1b	Physical properties and accelerated aging of conventional SMCs	8/31/05	3/31/06	
1.1b	Explore optimal extraction conditions for softwood chips	12/31/05	12/31/05	
2.2	Produce biobased SMCs	12/31/05	9/30/06	
1.2b	Complete softwood chips extraction and cooking with profiling digester	8/31/06	9/30/06	
1.3b	Complete the deposition study of hardwood hemicelluloses on kraft pulp	8/31/06	9/30/06	
1.4b	Complete oxygen delignification of soft wood kraft pulp with adsorbed hemis	8/31/06	9/30/06	
1.5	Complete bleaching of softwood and hardwood oxygen delignified pulp	8/31/06	9/30/06	
1.7	Go/No Go decision for feasibility study of hemi extraction technology at mill	8/31/06	9/30/06	IP will decide based on data
2.2	Complete SMC composites evaluation	3/31/07		
1.6	Completion of mass balance analysis of optimized and integrated process	5/31/07		
1	Experimental confirmation of integrated hemi extraction for soft and hardwood	5/31/07		
1+2	Complete final report - End of project	8/31/07		

Budget Data (06/30/06):

Quarter	From	To	Estimated Federal Share of Outlays*	Actual Federal Share of Outlays	Estimated Recipient Share of Outlays*	Actual Recipient Share of Outlays	Cumulative
4Q04	Start	12/31/04	$84,000	39,140.63	$25,000	18,522.38	$57,663.01
1Q05	1/1/05	3/31/05	$33,314	33,412.19	$40,524	12,263.43	$45,675.62
2Q05	4/1/05	6/30/05	$33,314	44,088.23	$40,524	8,645.04	$52,733.27
3Q05	7/1/05	9/30/05	$33,315	74,267.43	$40,524	2,290.42	$76,557.85
4Q05	10/1/05	12/31/05	$81,368	42,141.98	$39,702	32,870.90	$75,012.88
1Q06	1/1/06	3/31/06	$41,367	56,990.74	$39,702	49,640.83	$106,631.57
2Q06	4/1/06	6/30/06	$41,367	56,080.17	$39,702	23,866.58	$79,946.75
3Q06	7/1/06	9/30/06	$41,367		$39,702		
4Q06	10/1/06	12/31/06	$26,793		$29,703		
1Q07	1/1/07	3/31/07	$26,793		$29,703		
2Q07	4/1/07	6/30/07	$26,793		$29,703		
3Q07	7/1/07	9/30/07	$26,793		$29,703		
Totals			**$496,584**	**$346,121.37**	**$424,192**	**$148,099.58**	**$494,220.95**

Highly Energy Efficient Directed Green Liquor Utilization (D-GLU) Pulping

Lucia: NCSU

GO14308

QUARTERLY PROGRESS REPORT

Project Title:	HIGHLY ENERGY EFFICIENT D-GLU (DIRECTED GREEN LIQUOR UTILIZATION) PULPING
Covering Period:	April 1, 2006 through June 30, 2006
Date of Report:	30 July 2006
Recipient:	North Carolina State University (NCSU)
Award Number:	DE-FC36-04GO14308
Subcontractors:	Georgia Institute of Technology (GIT), Evergreen Pulp, Inc.
Other Partners:	N/A
Contact:	Lucian A. Lucia, 919.515.7707 (W), lucian.lucia@NCSU.edu
Project Team:	Dimitris Argyropoulos (NCSU), Sujit Banerjee (GIT), Rod Ledbetter (Evergreen Pulp, Inc.), Joe Springer (DOE)
Upload Site:	https://www.eere-pmc.energy.gov/SubmitReports.aspx

Project Objective & Update: This project will implement a green liquor (GL) pretreatment kraft pulping process at Evergreen Pulp, Inc. in Samoa, CA. We have allocated a significant sum of the current total yearly funding to Evergreen Pulp to promote the trial. Currently, Evergreen Pulp is undergoing a PSD Review that will delay the trial until the latter half of the year, but engineering plans for the implementation are virtually complete. Mill simulation research is being conducted to provide the best case scenario for the actual mill trial. It is anticipated that we will be following a careful UC extraction methodology in which increasing amounts of GL up to about 150 GPM will replace WL whose overall capacity will be reduced both in the UC & in the lower cooking zone. Laboratory work at NCSU is showing that during GL impregnation the lignin in the pulp has been enriched in Sulfur content that essentially makes it more "P*ulpable*."

STATUS AS OF 25 JULY 2006

MILESTONE 4: Answers to the economic use and application of additive-enhance GL pulping

- Imaging work showed that the diffusion of hydroxide lagged behind sulfide for redwood as previously observed with other species.
- The diffusion of both sulfide and hydroxide ions was very slow for black liquor but increase in 1:1 BL:WL. Diffusion of thiourea was much slower than that of sulfide.
- Diffusion profiles of hydroxide and sulfide were similar at 25 and 100°C indicating that the profiles were not influenced by acids liberated at the higher temperature.
- The yield of synthetic thiourea prepared in industrial (rather than synthetic) green liquor was lower than expected.
- Baseline analyses were conducted for digester liquor and pulp samples from Evergreen.

Diffusion of Sulfide and Hydroxide Ions in White or Green Liquor

Redwood from Evergreen Pulp was water-saturated and immersed in synthetic white liquor at 103°C under atmosphere pressure. Wood chips were periodically sampled was taken and split into two pieces. One was stained with 1% phenolphthalein (in ethanol) and the other was imaged with 3% silver nitrate solution. The results, shown in Figure 1, demonstrates that sulfidelags behind hydroxide as noted before with other wood species.

Saturated loblolly pine chips were also used for measurements at 103°C in green liquor with the following composition: Na_2S 38.87 g/L; NaOH 9.04 g/L; Na_2CO_3 76.94 g/L. As with white liquor, the diffusion of hydroxide lags behind that of sulfide as shown in Figure 2.

Figure 1: Ingress of sulfide (left) and hydroxide (right) in white liquor.

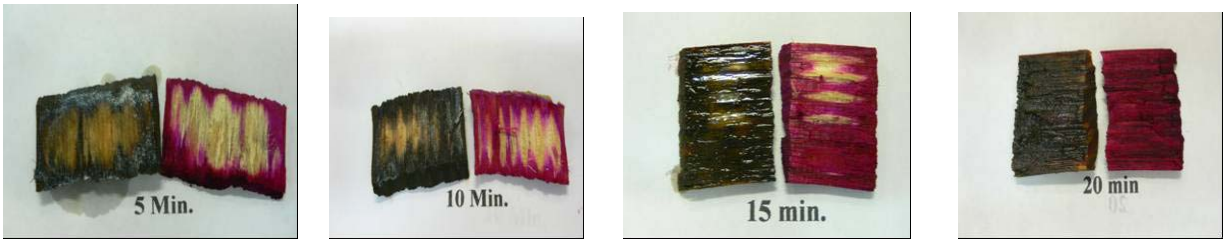


Figure 2: Ingress of sulfide (left) and hydroxide (right) in green liquor.

Diffusion of Sulfide and Hydroxide Ions in Black Liquor

Black liquor from the UC from Evergreen Pulp's digester was used. The liquor contained 13.16% solids and 10.07g/L of Na_2S and 4.37g/L of NaOH. Saturated sweetgum chips were used; the experiment was run at 103°C. As shown in Figure 3, the sulfide diffuses faster than hydroxide, although the rates of both ions are much slower than those in white or green liquor. In fact, the penetration is far from complete even after 1 hour. To confirm that the difference arose from differences in alkalinity, measurements were made in 1:1 BL:WL. Sweetgum woodchips were immersed at 103 °C as above. The results, illustrated in Figure 4, demonstrate that diffusion speeds up in the presence of WL.

Diffusion of Thiourea

An alkali liquor consisting of 3% thiourea, 41.9 g/L sodium hydroxide and 153.9g/L Na_2CO_3 was prepared. Sulfide ion was omitted from this liquor to avoid interferences in detection, since thiourea gives the same color upon staining with silver nitrate. The results shown in Figure 5 indicate that thiourea diffuses much more slowly than does sulfide. This could be because any charge exclusion experienced by sulfide would be absent in the thiourea. However, the larger molecular volume of thiourea would also reduce the diffusion rate.

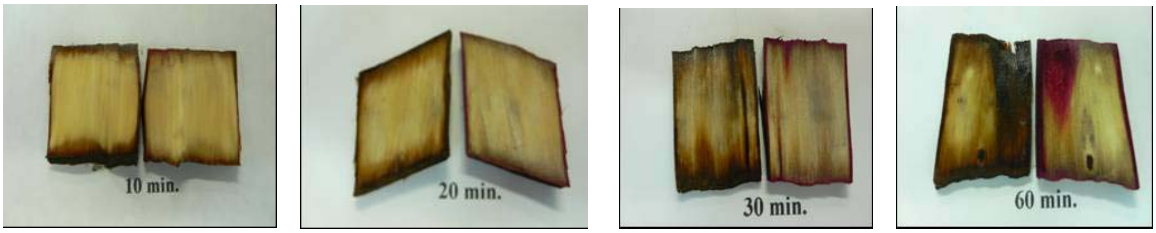


Figure 3: Ingress of sulfide (left) and hydroxide (right) in black liquor.

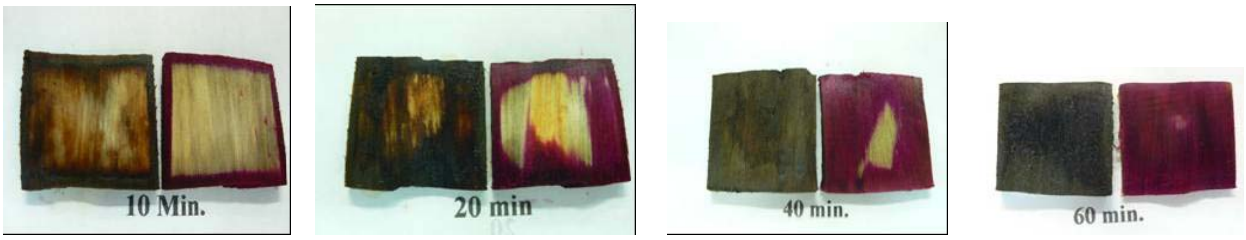


Figure 4: Ingress of sulfide (left) and hydroxide (right) in 1:1 black:white liquor.

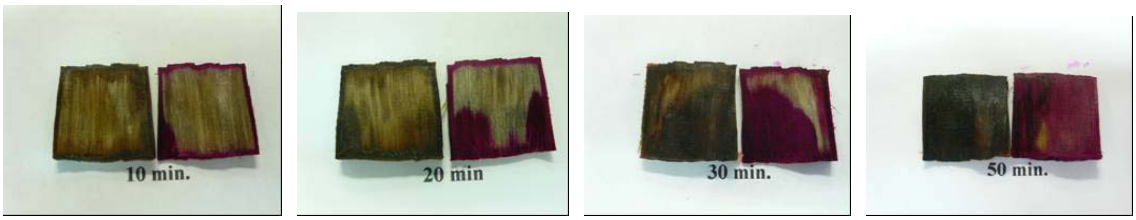


Figure 5: Ingress of thiourea (left) and hydroxide (right) in 1:1 black:white liquor.

Diffusion of Sulfide and Hydroxide Ions in White Liquor at Room Temperature

Some of the hydroxide will be consumed by wood acids as it enters the chip. However, this loss should not slow down the hydroxide diffusion front appreciably. The phenolic groups add up to 0.00025 moles per gram of oven-dry wood. Even if they were all accessible to the hydroxide and available to react (which is unlikely), they would consume only about 6% of the hydroxide diffused into the wood. In order to demonstrate that wood acids were not created during the time frame of our measurements, experiments were also run at room temperature. The same difference between the hydroxide and sulfide profiles was seen as shown in Figure 6. If acid generation had been significant, then more acid would have been generated at the higher temperature and the ingress of hydroxide would have been further retarded.

Use of Other Indicators for Imaging Hydroxide

Several indicators that change color at a pH higher than that appropriate for phenolphthalein were screened. These included Poirrier (from Wako Chemicals), tetryl (methyl-2,4,6-trinitrophenylnitramine, from Aldrich) and alizarin yellow R. Among those, the alizarin provides the best result. Staining of the cooked wood chips with this indicator provides an excellent contrast between the chemical diffused area and non-diffused area. Figure 7 displays two stained wood chips placed in white liquor at 103^{o}C. It clearly demonstrates that indicator stained area increases with the cooking time. Unlike phenolphthalein that changes color at pH 8.5, yellow R transitions at a pH above 10. It thus provides a different endpoint than that for pheolphthalein.

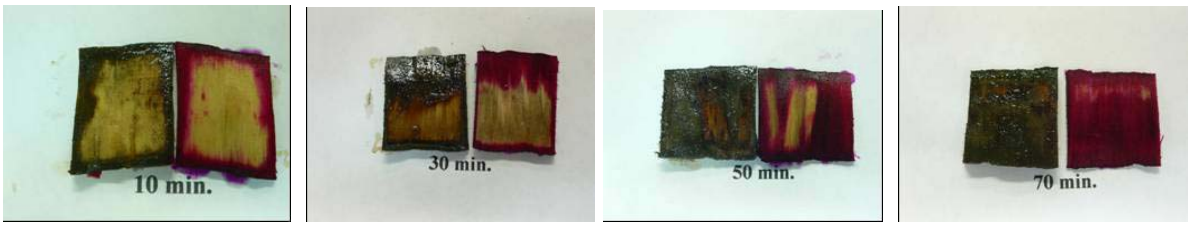


Figure 6: Ingress of sulfide (left) and hydroxide (right) in white liquor at room temperature.

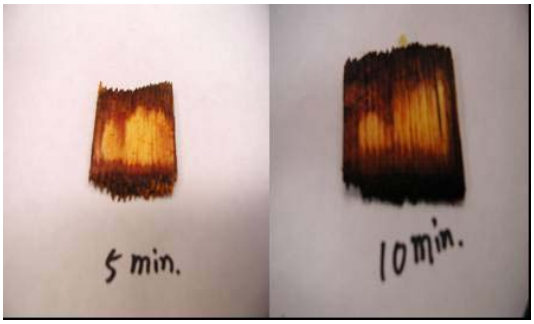


Figure 7: Pine chips stained with alizarin yellow.

Preparation of Thiourea for Lab Pulping

As discussed in the previous report, thiourea is readily generated from synthetic white liquor through a reaction between sodium sulfide and cyanamide. Three batches of thiourea were prepared from industrial green liquor (from Inland, Rome) in 1 liter/batches. In two of the three batches, the reaction was allowed to proceed for five hours, and the input of carbon dioxide was stopped at a pH of 9.6. The concentration of thiourea in both batches was about 7.3, as comparing to 14.0 g/L value obtained previously at a smaller (50ml) scale. The pH value of the third batch was allowed to drop to 8.6 before the carbon dioxide was stopped. Here, the thiourea concentration reached 10.4 g/L. But the chemical seemed to be unstable in this batch because a repeat measurement of thiourea concentration made the next day indicated that it had dropped to 5.3 g/L. The reason for the low thiourea yield may be due to impurities present in the industrial green liquor, since a significant amount of a greenish precipitate was observed in the reacted mixture. ICP analysis showed a sodium/sulfur ratio of 3/2 by weight.

Evergreen Mill Support

Support for a mill trial at the Evergreen mill Samoa, CA consisted of acquiring baseline data of the continuous digester operation and modeling liquor and component flows. Analysis of liquors from selected points was done to determine dissolved lignin content and total organic carbon as well as NaOH, Na_2S, and Na_2CO_3. These results are shown in Table 1. A similar analysis was done on samples collected in 10/05 and these were reported in the 4th Quarter, 2005 report. The calcium content of white and green liquors is also being measured to determine the potential for scaling.

Flow and component balances were determined for primary filtrate and liquor flows around the digester. This data will be incorporated into a model that should provide some predictive capability when green liquor is substituted for the white liquor.

Discussions with the Evergreen mill are on-going to formulate a trial plan and action criteria that will not comprise the operation of the digester. This will be crucial for the mill operators to understand the trial objectives. Additional lab pulping runs will be done with a series of cooks using Evergreen chips and liquors to measure the effect of green liquor substitution on K no. and to provide initial operating targets for the trial plan.

Table 1: Analysis of Evergreen Digester Liquor and Pulp Samples

	Sample	White Liquor	Top Circulation	Upper Cook Circulation	Main Extraction	Digester	BSW First Filtration
Liquor Analysis	**Units**						
Total Suspended Solids	mg/L as received	78	1528	20	44	1968	28
Total Dissolved Solids	mg/L as received	273568	141764	113416	209232.0	162788	91392
Total Solids	mg/L as received	273646	143292	113436	209276	164756	91420
Total Solids	% as received	27.20	10.79	13.16	19.00	15.42	8.86
Liquor density	g/mL	1.10	1.07	1.06	1.09	1.07	1.05
Residual Lignin (spectrophotometry)	% t.s.		9.9	26.3	45.1	45.1	45.7
Lignin	g/L		14.2	29.8	94.4	74.3	41.8
Hydroxide as NaOH (SCAN method)	mg/kg t.s.	351603	272245	35145	31559	18573	9156
NaOH	g/L Na_2O	74.57	30.23	3.09	5.12	2.37	0.65
Sulfide as $S^{=}$ ($AgNO_3$ titration)	mg/kg t.s.	55746	61428	33333	24737	12543	9094
Na_2S	mg/kg t.s.	135882	149731	81249	60296	30574	22167
Na_2S	g/L Na_2O	29.6	17.1	7.3	10.0	4.0	1.6
AA	g/L Na_2O	104.1	47.3	10.4	15.2	6.4	2.3
EA	g/L Na_2O	89.3	38.8	6.8	10.1	4.4	1.5
% Sulfidity	% AA	28.4%	36.1%	70.3%	66.2%	62.8%	71.3%
Total Carbon as C (coulometry)	% t.s.		11.7	27.8	36.6	36.6	47.7
Carbonate Carbon as C (coulometry)	% t.s.	0.40	2.13	1.52	1.16	1.17	1.33
Total Organic Carbon as C (coulometry)	% t.s.		9.57	26.28	35.44	35.43	46.37
Total Organic Carbon as C (coulometry)	g/L		13.71	29.81	74.17	58.37	42.39
Carbonate as $CO_3^{=}$ (coulometry)	% t.s.	2.0	10.7	7.6	5.8	5.8	6.6
Na_2CO_3	g/L Na_2O	5.7	15.8	8.9	12.5	9.9	6.2
CE	%	92.9%					
TTA	g/L Na_2O	109.8	63.1	19.3	27.7	16.3	8.5
Pulp Analysis							
Digester sample	Kappa No.	56.8					
BSW Sample	Kappa No.	28.8					
n.d. = not determined							
t.s. = total solids							

Lignin Work & Requirements for Mill Trial

In our efforts to better understand the effects of green liquor impregnation on the lignin structure, wood chips were impregnated with green and white liquors (white liquor , WL, refers to a liquor with the same compositions of green liquor, GL, but without carbonate) at 130^0C for 30 min. The lignin fraction was isolated from the treated-wood chips and analyzed by ^{31}P-NMR. These efforts were aimed at simulating the reactions of GL with lignin under conditions closer to a "real" impregnation stage. In this way, the diffusion of such liquors into wood was also taken into account. The yields of lignin isolated from wood chips treated with green and white liquors were quite similar (49% and 50% after white and green liquor treatments, respectively) as were the yields of lignin recovered from the black liquors generated during the wood impregnation (approximately, 5% in both cases). Characterization of these lignins showed no structural differences between white and green liquor treatments. A reduction of almost 20% in the total β-O-aryl ether linkages was observed in both cases. Overall, our work with isolated lignin (EMAL) or with wood chips have not shown any notable differences in reactivity between the WL and GL pretreatments.

Our reactivity studies have revealed another very important finding. *Pretreated EMALs were found to contain a significant amount of elemental sulfur bound to them by an as yet unknown mechanism*.

The total amount of sulfur bound with the lignin after a GL treatment was always found to be greater (by about 20%) than after a WL treatment, indicating that the carbonate plays a significant role in the sulfur incorporation. For this purpose, we have now devised an NMR based methodology for the precise quantification of the associated sulfur in WL and GL pretreated EMALs. Our measurements so far indicate that the reaction temperature has a significant affect on the sulfur uptake despite the pretreatment. At this point our attention is focused toward understanding and deriving the optimum conditions for maximum sulfur uptake at the pretreatment stage, since this local sulfidity is known to enhance the overall delignification during a subsequent kraft delignification stage.

Progress at Evergreen Pulp, Inc, Mill Facilty

EVERGREEN PULP INC.

- PSD Review currently underway which will be completely in 4 or less months
- Engineering plans for installation nearing completion
- Currently working with IPST & NCSU to finalize the digester liquor flows

Patents: Filed provisional patent for new method to extract hemicellulose from wood. In the midst of determining if our activated chemical intermediate is patentable.

Publications/Presentations (2005-2006):

1. Fundamental Considerations for Green Liquor Pretreatment Technologies. 2005 AIChE Conference (Atlanta, GA, April 11-14, 2005). Lucia, L.A.; Ban, W.; Argyropoulos, D.S.
2. Understanding Lignin During Pulping and Bleaching. 2005 AIChE Conference (Atlanta, GA, April 11-14, 2005). Lucia, L.A.; Argyropoulos, D.S.
3. Liu, Q.; Singh, J.M.; Wang, S.; Ban, W.; Lucia, L.A. "The Influence of Green Liquor and AQ-Modified Kraft Pulping on Fiber Hexenuronic Acid and Carboxyl Group Content for Linerboard Grade Pulp." *Cell. Chem. Technol.* **2005**, *35*, 483-498.
4. Ban, W.; Lucia, L.A. "Kinetic Profiling of Green Liquor Pretreatment." *Ind. Eng. Chem. Res.* **2006**.
5. Lucia, L.A. "A Quantitative Comparison of the Kinetic Features of Green Liquor-Modified Versus Conventional Kraft Pulping," 2005 TAPPI Engineering, Pulping, and Environmental Conference (Philadelphia, PA, August 28031, 2005). Lucia, L.A.; Argyropoulos, D.S.; Ban, W.; Courchene, C.
6. Lucia, L.A. "A Quantitative Comparison of the Kinetic Features of Green Liquor-Modified Versus Conventional Kraft Pulping," Wood and Paper Science Departmental Graduate Seminar Series (Raleigh, NC, 19 January 2006).
7. Banerjee, S.; Lucia, L.A.; Jacobson, A. "Green Liquor Pretreatment Pulping: Importance of Diffusion." TAPPI High Yield Symposium: Growing Yield from the Ground Up. Atlanta, GA; May 16-18, 2006.

MILESTONE STATUS TABLE:

ID Number	Task / Milestone Description	Planned Completion	Actual Completion	Comments
1	Organization of pulping project mill consortium and agenda for implementation over four years	01/01/05	Currently pursuing: have interest from Aracruz, Stora-Enso, Longview Fibre, International Paper, Inland, Lincoln Tissue & Paper	This consortium will be at NCSU and leverage work of DOE
2	Addressing the necessary research tasks to address mill implementation requirements at Evergreen Pulp, Inc.	08/15/05	04/01/2006	In progress with Andrew Kulchin at Evergreen Pulp, Inc.
3	Green liquor (GL) laboratory pulping studies associated with Task 2	12/01/05	04/15/2006	Baseline work has been completed to satisfaction of mill
4	**MILESTONE 1: Answers to feasibility and planning of GL pulping process (pulp properties, secondary effects on mill) in target mill**	01/15/06	04/15/2006	We have addressed this as well as we will do at this point
5	Fundamental and applied determination of handling green liquor impregnation and liquor flows; work in coordination with Milestone 4 to eliminate any problems in mill; have supporting engineering design	06/30/06		We are almost nearing the completion of this task since we are receiving the engineering design plans
6	Finalization for mill trial and implementation	07/01/06		Delayed by about 4 months
7	**MILESTONE 2: Answers to the question of how the build up of scale in the pulp mill and downstream occurs**	12/31/06		This milestone is not critical at this point – our work does not point to it being a "show stopper"
8	**MILESTONE 3: Answers to the possibility of significantly altering green liquor recausticization based on Task 5**	06/01/07		This is potentially a new avenue for used GL to be recycled – currenly not a high priority
9	Mill trial	07/01/07		The PSD Review has pushed this task back until the end of 2006

10	Applied and fundamental studies for the pulping catalyst including role, fate, impact on pulp properties, impact on recovery, exploration for alternative organics	12/31/07	The reset of the tasks from this point on are part of phase 2 GL pulping
11	**MILESTONE 4: Answers to the economic use and application of additive-enhance GL pulping**	02/01/08	
12	Selection of mill(s) that has conducted the appropriate mill studies for additive-enhanced GL pulping	04/01/08	Evergreen Pulp may be one of the potential candidates
13	Exploration of mill specific issues related to odor, environmental issues, solid impacts, and corrosion for Milestone 4	06/30/08	
14	Final Engineering report and lab work supporting mill trials for additive-enhanced GL impregnation	12/31/08	
15	**MILESTONE 5: Final research report to consortium on implementation plan for additive-enhance GL impregnation**	01/31/09	
16	Mill trial at selected sites based on engineering criteria developed with mill	09/30/09	
17	Final report for the industry and DOE	12/31/09	

Budget Data (as of date): The actual spending should reflect the money actually spent on the project in the corresponding periods.

524402 Quarterly Report Through 07/30/2006

To	Estimated Federal Share of Outlays	Actual Federal Share of Outlays	*Estimated Recipient Share of Outlays	Actual Recipient Share of Outlays	Cumulative
9/30/2004	-	-		-	-
12/31/2004	-	-		-	-
3/31/2005	750	750.41	5,935	5,935.26	6,685.67
6/30/2005	11,855	37,713.92	5,920	5,757.81	43,471.73
9/30/2005	183,900	168,540.18	2,988	2,987.55	171,527.73
12/31/2005	79,900	86,152.73	12,392	12,391.61	98,544.34
3/31/2006	127,995	124,301.84	4,879	4,879.43	129,181.27
6/30/2006	**139,210**	**26,988.22**	**41,580**	**41,579.51**	**68,567.73**
9/30/2006	139,960		733,655		-
12/31/2006	139,960		733,655		-
3/31/2007	139,960		9,549		-
6/30/2007	139,960		9,549		-
9/30/2007	139,960		9,548		-
12/31/2007	139,960		9,948		-
3/31/2008	358,831		10,000		-
6/30/2008	358,730		10,000		-
9/30/2008	358,730		10,000		-
12/31/2008	358,505		10,000		-
	2,818,166	444,447.30	1,619,198	73,531.17	517,978.47

* Updated quarterly

A Novel Alkali Fractionation Technology to Separate Wood Components

Lehrburger: PureVision

GO15154

Progress Report #4, July 31, 2006

PureVision Technology, Inc.

Financial assistance award DE-FG36-05GO15154

Project:	Fractionation of Loblolly Pine Woodchips into Pulp used for Making Paper Products
DOE award #:	Financial assistance award DE-FG36-05GO15154
Recipient:	PureVision Technology, Inc.
PureVision office:	511 McKinley Ave, Ft. Lupton CO 80621
PureVision lab:	Hazen Research Inc., 4601 Indiana, Golden CO 80403
Project Director:	Ed Lehrburger, 303-857-4530, Ed@purevisiontechnology.com
PureVision P.I.	Kiran Kadam PhD, 303-279-4502 X 354, Kiran@purevisiontechnology.com
Reporting period:	April 1, 2006 – June 30, 2006
Date of report:	July 31 2006
Report by:	Kiran Kadam, Ed Lehrburger
Subcontractors: (this period)	International Paper Company (cost share only)*
DOE Project Team:	Beth H. Dwyer, Contracting Officer, Golden Field Office Joe Springer, Project Manager Tim Ramsey, Project Monitor

Progress Report #4
Narrative

Project Objectives:
Perform fractionation studies using the PureVision process development unit (**PDU**) on loblolly pine; evaluate the qualities of the cellulosic fraction; demonstrate use of the technology for processing woodchips into fiber; and to undertake preliminary economic studies.

Background:
During this reporting period, fractionation studies on loblolly pine were completed. The modifications made to the PDU, approved by DOE under award #DE FG 36 GO85004, have proved to be beneficial to the DE FG-36-05-GO15154 program.

Status:
Most of the primary feedstock processing and fiber analytical work for this program were completed during this reporting period. We conducted pretreatment experiments using the PDU configured for a single stage ethanol extraction process. We studied the effect of different process parameters (i.e., temperature, NaOH concentration, and residence time) and, based on the best delignification results, we selected two sets of conditions for campaign runs. Campaigns were run at combinations of low NaOH concentration–long residence time and high NaOH concentration–short residence time. Fiber samples (2 lb dry basis) were collected and sent to International Paper (IP) for analysis. To date, IP has provided most of the critical data to PureVision to complete the fiber quality analysis report. This data will serve as a foundation for completing marketing analysis. PureVision also conducted enzymatic hydrolysis experiments on the fiber samples. The results are being analyzed.

All DOE funds for the program have been exhausted and some of the program tasks will not be completed. These tasks include handsheet analyses, microscopic analyses and economic modeling tasks. All loblolly pine feedstock-processing tasks have been completed. Remaining project tasks include completing analytical work, a preliminary marketing assessment and presenting the technical report.

Plans for next quarter:
During the next quarter (July 1, 2006 through September 30, 2006), PureVision will undertake the following:

Task 2. Complete the fiber assessment report. PureVision and IP personnel will discuss all data generated during the program and complete a fiber assessment report. PureVision will complete the fiber assessment report; however subtask 2.2, microscopic evaluations, will not be completed due to lack of funds. Also thus far IP has not undertaken Subtask 2.3, handsheet testing. IP and PureVision will discuss if IP desires to pursue this and collectively will determine if any more work will be completed by IP on producing and testing handsheets. Subtask 2.4, a fiber quality analysis report, is planned to be completed by September 1.

Task 3. Market assessment. This work will be addressed in August. We plan to begin subtask 3.1, market assessment, once we have completed our review of the data from Task 2, expected around mid-August. The Task 3 assessment work will continue through late August when we expect to complete subtask 3.2, presenting the results of the market assessment.

Change in Program: The following changes to the program are noted:

1. A scheduled trip by PureVision's Paul Mann to IP to undertake microscopic testing was postponed by IP. The trip, scheduled May 21 and 22, 2006, was cancelled by IP due to scheduling conflicts with other corporate programs. As a result microscopic work on the fiber samples will not be completed.
2. IP may not complete handsheet testing as part of the fiber quality-testing program.
3. PureVision will not complete the economic modeling task, as funds for the program have been exhausted.

Patents: No patents were applied for or resulted from the award to date.

Publications/Presentations: None to date.

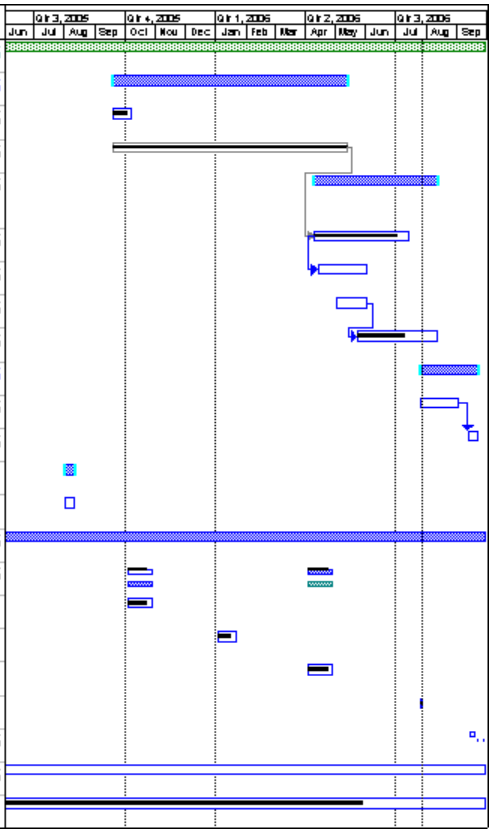
Milestone Status Table:
A task-tracking chart is included below in addition to an updated Gantt chart for the project.

We expect to complete the project by September 30, 2006. The revised project schedule is shown below.

ID Number	Task / Milestone Description	Planned Completion	Actual Completion	Comments
1	PureVision: Loblolly Pine-to-Pulp			
1.1.	Fractionate wood chips	5/12/06	6/15/06	Complete
1.2.	Fiber Assessment			
1.2.1	IP Screening Experiments	6/2/06	6/30/06	Nearly complete
1.2.2	Microscopic evaluations	6/2/06		No scheduled
1.2.3	IP Handsheet tests	6/2/06		Not scheduled
1.2.4	Complete FQA report	6/12/06		New end date: 8/15/06
1.3.	Market Assessment			
1.3.1	Market Assessment	6/16/06		New end date: 8/30/06
1.3.2	Present results	6/23/06		9/30/06 in final report
1.4.	Economic modeling			
1.4.1.1	Review data, create model	6/5/06		Not scheduled
1.4.2	Economic modeling	6/19/06		Not scheduled
1.5	Project monitoring, reporting			
1.5.2	Prepare project final report	6/30/06		9/30/2006

Updated Gantt Chart: (as of June 30, 2006)

ID	Task Name	Start	Finish
0	PureVision Loblolly Pine-to-Pulp	Wed 6/1/05	Fri 9/29/06
1	1 Fractionate wood chips	Mon 9/19/05	Fri 5/12/06
2	1.1 Receive feedstock	Mon 9/19/05	Fri 10/7/05
3	1.2 Process feedstock	Mon 9/19/05	Fri 5/12/06
4	2 Fiber Assessment And Microscopic Evaluation	Mon 4/10/06	Fri 8/11/06
5	2.1 IP Screening Experiments	Mon 4/10/06	Fri 7/14/06
6	2.2 Microscopic Evaluations	Thu 4/13/06	Fri 6/2/06
7	2.3 IP Handsheet Tests	Mon 5/1/06	Fri 6/2/06
8	2.4 Complete Fiber Report	Mon 5/22/06	Fri 8/11/06
9	3 Market Assessment	Mon 7/24/06	Fri 9/22/06
10	3.1 Market assessment	Mon 7/24/06	Fri 9/1/06
11	3.2 Present joint market report	Mon 9/11/06	Fri 9/22/06
12	4 Economic Modeling	Mon 8/1/05	Thu 8/11/05
13	4.1 Task cancelled, lack of funds	Mon 8/1/05	Thu 8/11/05
14	5 Monitor project &reporting	Wed 6/1/05	Fri 9/29/06
15	5.1 Prepare quarterly reports	Mon 10/3/05	Fri 7/28/06
16	5.1.1 Prepare quarterly reports	Mon 10/3/05	Fri 10/28/05
17	5.1.2 Prepare quarterly reports	Tue 1/3/06	Fri 1/20/06
18	5.1.3 Prepare quarterly reports	Mon 4/3/06	Fri 4/28/06
19	5.1.4 Prepare quarterly reports	Mon 7/24/06	Fri 7/28/06
20	5.2 Prepare final report	Tue 9/12/06	Fri 9/29/06
21	5.3 Manage project	Wed 6/1/05	Fri 9/29/06
22	5.3.1 Manage project	**Wed 6/1/05**	**Fri 9/29/06**



Budget Data (as of 06/30/06): The actual spending should reflect the money actually spent on the project in the corresponding periods.

Project Spending and Estimate of Future Spending							
Quarter	**From**	**To**	**Estimated Federal Share of Outlays**	**Actual Federal Share of Outlays**	**Estimated Recipient Share of Outlays**	**Actual Recipient Share of Outlays**	**Cumulative**
Est. cost share	Start	9/30/04				$150,000	$150,000
4Q04	10/1/04	12/31/04					
1Q05	1/1/05	3/31/05					
2Q05	4/1/05	6/30/05					
3Q05	7/1/05	9/30/05	$ 50,676	$ 37,718	$ 0		$37,718
4Q05	10/1/05	12/31/05	$ 54,303	$ 9,542	$ 0		$47,260
1Q06	1/1/06	3/31/06	$ 21,085	$ 25,523	$ 0		$72,783
2Q06	4/1/06	6/30/06	$ 23,936	$77,217	$ 0	$72,580	$222,580
3Q06	7/1/06	9/30/06					
4Q06	10/1/06	12/31/06					
1Q07	1/1/07	3/31/07					
2Q07	4/1/07	6/30/07					
3Q07	7/1/07	9/30/07					
4Q07	10/1/07	12/31/07					
Totals			**$150,000**	**$ 150,000**	**$0**	**$222,580**	**$380,341**

Yield Improvement and Energy Savings Using Phosphonates as Additives in Kraft Pulping

Tschimer: University of Minnesota

ID14433

QUARTERLY PROGRESS REPORT

Project Title: Yield Improvement and Energy Savings Using Phosphonates as Additives in Kraft pulp

Covering Period: 1/1/06-3/30/06

Date of Report: July 28, 2006

Recipient:
University of Minnesota
Office of Sponsored Projects
McNamara Alumni Center
200 Oak Street SE, Room 450
Minneapolis, MN 55455
Congressional District:5

Award Number: DE-FC36-03ID14433

Subcontractors: none

Other Partners:
Solutia Inc. (Company)
Cost sharing, in-kind contributions of salary, travel, chemicals and supplies at $15,000/year
Sheldon Verrett, 385 Marshall Avenue, Webster Grove, MI 63119
Tel. (312) 872-2127

Contact(s):
Ulrike Tschirner (612) 624-8798, e-mail : ulrike@umn.edu
Timothy Smith (612) 624-6755, e-mail: smith463@umn.edu

Project Team:
DOE-HQ contact: Gibson Asuquo Project Officer
Beth Dwyer, Financial Assistance Officer
Sheldon Verrett (Industry contact)
Sharon Bartlett (U of Mn grant and Contract Administrator)

Project Objective: Develop a commercially viable modification to the Kraft process resulting in energy savings, increased yield and improved bleachability. Evaluate the feasibility of this technology across a spectrum of wood species used in North America. Develop detailed fundamental understanding of the mechanism by which phosphonates improve KAPPA number and yield. Evaluate the North American market potential for the use of phosphonates in the Kraft pulping process. Examine determinants of customer perceived value and explore organizational and operational factors influencing attitudes and behaviors. Provide an economic feasibility assessment for the supply chain, both suppliers (chemical supply companies) and buyers (Kraft mills). Provide background to most effectively transfer this new technology to commercial mills.

Background: The project is to advance the development, optimization, and potential commercialization of a new Kraft pulping process using phosphonates as additives that promises opportunities for substantial energy savings, increased yield, and bleaching cost reduction. Our preliminary studies suggest energy savings in the digester of up to 10%, increased yield of 4-6%, increased ratio of delignification and improved pulp strength. As the predominant pulping technology worldwide for producing chemical pulps of high strength, the

Kraft process is used to produce approximately 51 million metric tons of pulp per year in the US. The approach of using phosphonates as additives in the Kraft cook is entirely new. Phosphonates are acids or salts with highly anionic charges, containing at least one functional group, $-P(=O)(OH)_2$, attached to a carbon atom. Their complex structures are characterized by phosphoryl bonds and multiple metal ion chelating sites. Phosphonates are soluble and stable in aqueous systems to high temperatures, pressures and extreme pH values. Phosphonates have surfactant and dispersing properties and are expected to facilitate penetration of chemicals into chips as well as prevent re-deposition of lignin. In addition their chelating properties are expected to favor brightness improvements in subsequent bleaching processes since bleaching sequences such as oxygen, chlorine dioxide or peroxide are negatively influenced by presence of transition metals. Preliminary experiments performed in our lab far exceeded our expectations with respect to increased lignin removal, improved yield, conservation of pulp viscosity and bleached brightness.

Status:
Evaluation of different wood species has been completed and summary of results was compiled (Table 1). Five different hardwood samples and four softwood samples have been evaluated with number of different phosphonate products. Aspen clearly shows largest response with respect to yield improvement and increased lignin removal. The remaining hardwood species did show some response, but yield improvement as well as increased lignin removal was less pronounced than for aspen. In most cases the phosphonate with the most significant impact was Dequest 2016 (HEDP, 1-Hydroxyethylene-1,1-di-phosphonic acid). This is the smallest of the phosphonates we used in our study. Softwoods showed no or very small improvement. For all wood species tested bleaching response for pulps produced with phosphonates in the digester showed significant improvement. This effect clearly is caused by the removal of metals from the pulp fiber. Unfortunately due to the price of phosphonates their use for improvement of bleaching alone might not be economically feasible.

We are continuing several other sections of this project. As reported earlier molecular weight of lignin fragments isolated from black liquor appears to be larger for cooks using additives. Our department is in the process to install a multi angle light scattering detector (not part of this proposal but funded through the university). We will be using this new equipment to complete this part of our study. Completion is expected by October.

We are continuing study on black liquor recirculation and are starting to summarize all data collected during this project.

Market Study:
While analysis continues on the survey data collected in August/September 2005, particularly around the structural equations modeling efforts, in this past quarter we have begun the secondary literature review and data collection required for the economic and energy assessment portion of the project.

The database of north American Kraft pulp mills developed in the beginning of this project (2003) is in the process of being updated to include revised and more accurate data regarding mill production, as well as the introduction of a number of additional fields driven by the

technical findings from the laboratory, most specifically the inclusion of estimated species mix utilized in mill production. In addition, previous research around energy consumption and engineering economics specific to the Kraft pulping process is underway. In the following months this data will be integrated with survey findings in an effort to identify specific organizational and operational factors related to adoption propensity to ultimately provide both micro- and macro- economic and energy performance estimates related to the adoption of this technology.

Plans for next quarter:
We will continue the examination of lignin fragments obtained from black liquor and will continue black liquor recirculation study. We will start compiling data for the final report in December. For the market study data will be integrated with survey findings in an effort to identify specific organizational and operational factors related to adoption propensity to ultimately provide both micro- and macro- economic and energy performance estimates related to the adoption of this technology.

Table 1:

Wood Species	**Phosphonates tested**	**KAPPA number reduction**	**Yield improvement at given KAPPA**	**Bleaching improvement at same starting KA**	**Comments**
Aspen	2016, 2086 2066, 2096 6004, 2054 7000, 3000	7 points (0.2%) 10 points (0.4%)	4-7%	2-3 points	Considerably better response than AQ, 2016 best performing
Alder	2016, 2086 2066, 2096 6004, 2054 7000, 3000	Up to 2 points at high KA	~ 1% at high KA	1.5 Points	AQ performed equal or better than any of the phosphonates
Birch	2016	3 points	1-2%	NA	
Maple	2016	Up to 4 points at higher KA	1-2 points	1 point	
mixed southern Hardwood	2016	Up to 1 point	1-2%		Similar in yield improvement than AQ, but less effective in increasing lignin removal
Pine	2016 2006	No change	1-2%	2-3 points	Yield same as AQ addition
Spruce	2016	No change	No change	2-3 points	
Fir	2016, 2086 2066, 2096 6004, 2054 7000, 3000	1 point at high AA	~ 2% for 2054	3 points	2054 better in yield than AQ
Mixed Softwood	2016, 2086 2066, 2096 6004, 2054 7000, 3000	No change	No change	2-3 points	

Milestone Status Table:

ID Number	Task / Milestone Description	Planned Completion	Comments
1	Evaluation of different wood species	4/1/06	completed
2	Characterization of fundamental background	12/31/06	On-going
2.1	Dispersion properties	10/1/03	Completed
2.2	Penetration into wood chips	4/1/04	Completed
2.3	Characterization of residual lignin	8/1/04	Completed
2.4	Characterization of black liquor lignin	10/01/04	Completed
2.5	Comparison of phosphonate responses	4/1/05	Completed
2.6	Optimization of process	10/1/05	Completed
2.7	Fate of phosphonates in the process	12/31/06	On-going
3.	Market study	12/31/06	On-going
3.1	Secondary Literature review	3/31/04	Completed
3.2	Model development	3/31/04	Completed
3.3	Interview with mill personal	3/31/04	Completed
3.4	Survey design and implementation	10/31/05	Completed
3.5	Economic feasibility assessment	10/31/06	40% completed
4.	Analysis and final report	12/31/06	60% completed

Approved Budget Data:

Phase / Budget Period			DOE Amount	Cost Share	Total
	From	To			
Year 1	4/1/03	3/31/04	$157,680	$ 41,145	$ 198,825
Year 2	4/1/04	3/31/05	$106,682	$ 42,190	$ 148,872
Year 3	4/1/05	12/31/06	$105,956	$ 43,277	$ 149,233
Year 4	NA				
Year 5	NA				
		Totals	$ 370,318	$ 126,612	$ 496,930

Project Spending and Estimate of Future Spending							
Quarter	**From**	**To**	**Estimated Federal Share of Outlays***	**Actual Federal Share of Outlays**	**Estimated Recipient Share of Outlays***	**Actual Recipient Share of Outlays**	**Cumulative**
	Start	9/30/2004	Note 1	157,930.00	Note 1	67,930.00	225,860.00
4Q04	10/1/2004	12/31/2004	21,900	179,830.00	8797.00	76,727.00	30,697.00
1Q05	1/1/2005	3/31/2005	25,855	205,855.00	8797.00	85,524.00	34,562.00
2Q05	4/1/2005	6/30/2005	22,713	228,397.00	8797.00	94,321.00	31,510.00
3Q05	7/1/2005	9/30/2005	27,164	255,561.00	8797.00	103,118.00	35,961.00
4Q05	10/1/2005	12/31/2005	19,984	275,545.00	8797.00	111,915.00	28,781.00
1Q06	1/1/2006	3/31/2006	29,045	304,590.00	8797.00	120,712.00	37842.00
2Q06	4/1/2006	6/30/2006	4,892	309,482	5900.00	126,612	10792.00
3Q06	7/1/2006	9/30/2006	32,000				32,000.00
4Q06	10/1/2006	12/31/2006	28,835				28,835.00
1Q07	1/1/2007	3/31/2007					
2Q07	4/1/2007	6/30/2007					
3Q07	7/1/2007	9/30/2007					
4Q07	10/1/2007	12/31/2007					
Totals			**370,318**	**309,482**	**126,612**	**126,612**	**496,930.00**

Design and Demonstration of Multiport Cylinder Dryers

Choi: Argonne National Laboratory

Agr id:11430

QUARTERLY PROGRESS REPORT

Project Title: Development and Full-Scale Demonstration of Multiport Dryer Technology for the Forest Products Industry.

Covering Period: April 1, 2006 through June 30, 2006

Date of Report: July 31, 2006

Recipient:
Argonne National Laboratory
Energy Systems Division, #335
9700 S. Cass Avenue
Argonne, IL 60439

Award Number: 49682

Subcontractors:
Ability Engineering
Design Solutions, Inc.

Other Partners:
The University of Illinois at Chicago
Kadant Johnson Inc. (formerly The Johnson Corporation)
International Paper

Contact(s):
Steve Choi
630-252-6439
630-252-5568 (fax)
choi@anl.gov

Project Team: DOE-HQ contact: Isaac Chan and Drew Ronneberg

Project Objective: The objectives of the proposed research are to design and fabricate a prototype multiport dryer and to conduct multiport dryer performance tests in a full-scale test dryer for retrofit applications.

Background: Argonne National Laboratory (ANL) has already developed a multiport dryer design concept that could create breakthroughs in drying pulp and paper. In a proof-of-concept test, ANL demonstrated the feasibility of this concept. A series of steam condensing tests in ANL's Multiport Dryer Heat Transfer Test Facility showed that the condensing heat transfer coefficient for multiport dryers is about 7 times greater than that in a conventional dryer with spoiler-bar enhancement and about 20 times greater than that in a conventional dryer without spoiler bars. Furthermore, the tests showed that dryer shell surface temperatures are more uniform in multiport dryers than in conventional dryers. With the

feasibility of the concept already proven in ANL's unique Multiport Dryer Heat Transfer Test Facility, the next step is to continue the project into full-scale tests that lead to commercial demonstration of the technology. At the same time, additional testing in the Multiport Dryer Heat Transfer Test Facility is scheduled to allow the development of predictive heat transfer relations for multiport dryers over a range of operating and design conditions.

Status: During the reporting period, the final design was completed of the Multiport Dryer insert to be tested in the Kandant-Johnson full scale steam dryer test facility. Based on the results of the previous installation of the insert into the Johnson shorted dryer, several areas were identified to ease installation in the full size unit. These areas have been incorporated into the final design. In addition, the results of the smoke test of the insert in the shortened dryer showed a connection area that needed modification to reduce the potential for steam by-pass. That too, has been addressed in the final design. During the reporting period, ANL worked with Kandant Johnson and Ability Engineering (the Multiport Dryer insert manufacturer) to arrive at the final insert design addressing these various issues. A decision was made to test the Multiport Dryer and Spoiler Bar technology simultaneously in the full-scale facility. Materials previously installed in the shortened dryer will be utilized, and additional panels will be manufactured to add channel length. When testing the Multiport dryer insert and spoiler bars simultaneously in the Kadant-Johnson facility, there is an option as to where to locate the two sections relative to the steam inlet and condensate outlet. That location has been resolved, and appropriate supports have been included in the final design to accommodate that placement. A contract is being written for the manufacturing of the additional panels and hardware for the final insert design discussed above. We have learned that Multiport dryer technology has earned a very prestigious R&D 100 Award for 2005. The award recipients are Stephen Choi, David France and Ralph Niemann from the ANL team, and Gregory Wedel and Gerald Timm from Kadant Johnson. This award seeks to recognize the 100 most innovative and potentially significant technical inventions world-wide on an annual basis. ANL is extremely pleased with this recognition of its achievement, and DOE sponsorship is recognized through it.

Plans for Next Quarter: The next step will be the manufacturing of the additional panels and hardware for the final insert design. This work will be followed by the full scale testing in the Kandant-Johnson dryer test facility scheduled for October 2006.

Milestone Status:

ID Number	Task / Milestone Description	Planned Completion	Actual Completion
	Conduct Full-Scale Test I		
1.	Complete the fabrication of a full-length Multiport Dryer (MD) insert.	9/15/05	
2.	Conduct full-scale MD test I.	9/30/05	
3.	Complete laboratory tests for the design of a prototype MD.	9/30/05	
	Conduct Full-Scale Test II		
1.	Modify the design of a MD insert as needed.	11/15/05	
2.	Fabricate a full-length MD insert.	11/30/05	
3.	Conduct full-scale MD test II.	12/31/05	
4.	Assess technical/economic feasibility of MD technology for retrofit application.	3/30/06	

Budget Data (as of 6/30/06):

		Approved Spending Plan			Actual Spent to Date		
Budget Period		DOE Amount	Cost Share	Total	DOE Amount	Cost Share	Total
From	To						
1/1/05	3/30/06	207	200	407	166	0	166
Totals		207	200	407	166	0	166

On-Line Fluidics Controlled Headbox

Aidun: IPST at Georgia Tech

GO10416

As of August 15, 2006, the PI has not yet submitted an updated status report for the quarter ending June 2006.

The Lateral Corrugator

Schaepe: IPST at Georgia Tech

GO10616

QUARTERLY PROGRESS REPORT

Project Title: An Improved Method of Manufacturing Corrugated Boxes: Lateral Corrugator

Covering Period: April 1, 2006 – June 30, 2006

Date of Report: August 07, 2006

Recipient: Institute of Paper Science and Technology at Georgia Tech
500 10th St, NW
Atlanta, GA 30332-0620

Award Number: DE-FC36-01GO10616 M3

Subcontractors:

Other Partners: Temple-Inland Paperboard and Packaging, Smurfit-Stone Container Corp., MarquipWardUnited, Corrugated Gear, Albany International, Armstrong, Container Graphics Corporation, CUE, Harper-Love Adhesives, The Johnson Corporation, Corn Products International, Hardy Instrumentation, WIKA Instrument Corp., Chicago Electric, Pamarco

Contact: Michael Schaepe, Senior Research Engineer, Principal Investigator
404-894-6640
michael.schaepe@ipst.edu

Perry Arrington, Robert Hall, Mark Szlemko, Mark Urbin

Project Team: Gibson Asuquo, gibson.asuquo@go.doe.gov
Tim Ramsey, tim.ramsey@go.doe.gov
Beth Dwyer, beth.dwyer@go.doe.gov

Project Objective: The goal of this project is to develop a commercially viable lateral corrugating process. This includes designing and building a pilot lateral corrugator, testing and evaluating the pilot machine, and developing a strategy for commercialization.

Background:

Since paper is non-isotropic and fibers tend to orient in the machine direction, machine direction (MD) compressive strength of paper exceeds that of the cross-machine direction (CD). In a conventional corrugator, the paper machine direction is perpendicular to the flute direction. Therefore, a typical corrugated container cannot take advantage of the stronger compressive strength of the paper machine direction.

Experiments conducted at IPST demonstrated that combined-board with the linerboard MD orientation in the transverse direction of the combined-board generated box compression strength improvements of 30% over conventionally oriented board. Yet, with the medium MD orientated conventionally, flat crush, handling toughness, and board rigidity were maintained. It was found that a box utilizing 15% lighter materials with the linerboard transversely oriented generated comparable stacking strength to a conventional box.

A method to produce combined-board with the linerboard oriented in the transverse direction has been considered. This method of box manufacturing could reduce fiber consumption 15% and improve the compressive strength to weight ratio of corrugated shipping containers considerably, thereby **significantly reducing energy usage both in manufacturing and transportation**. The technology to produce such a sheet would involve conventional fluting of the medium. The transverse orientation of the linerboard would be achieved through a sheeting operation. Single-facing and double-backing would again involve conventional but state-of-the-art corrugating technologies.

This project has been undertaken to construct a lateral corrugator and evaluate the resulting combined-board. The project will entail the development of a testing program, the design and construction of a pilot lateral corrugator, and the evaluation of conventional and lateral combined-board samples and boxes.

During the first year of this project two major program objectives were initiated. The lateral corrugator design was begun and experiments to explore the unique heat transfer opportunities of the lateral corrugator were undertaken. The design of the lateral corrugator proceeded as a retrofit to the newly completed double-backer at the IPST Industrial Engineering Center. The design incorporates a unique glue applicator system to allow the use of high solids adhesives and eliminates single-face festoons from the corrugating process. Both of these unique design features will **reduce the energy requirements** to produce combined-board. The heat transfer experiments aided in the selection of the post-heating elements to be used for the lateral corrugator.

The second year of the program focused on the fabrication and installation of the corrugating roll stack, the lateral corrugator drive system, the hydraulic and steam supply systems, and the glue machines. Also during the second year of the program the advantages of cut-to-width sheeting of lateral corrugator operation were investigated. Cut-to-width sheeting will reduce trim waste at the paper mill and box plant and simplify paper roll management. Since with lateral corrugating the paper is sheeted, matching paper-roll widths at the box plant would not be necessary and corrugator trim waste could be precisely controlled. For paper companies, the advantage of the lateral corrugator is very attractive since it would allow papermakers to reduce waste. With the cut-to-width capability of the lateral corrugator, paper companies could produce paper-roll widths to fully trim out paper machines. Also, papermakers could produce paper-rolls to optimize logistics, that is, maximize transportation efficiency. With these advantages energy saving would be considerable.

Future work will involve the integration of sheet feeders and splicing equipment into the lateral corrugator. The final result will be a pilot facility to produce lateral combined-board blanks of sufficient size to manufacture small boxes.

The IPST web page (http://www.ipst.gatech.edu/) includes information and pictures of the lateral corrugator. The lateral corrugator is highlighted under Research. To view the lateral corrugator program on the IPST web page, go to Corrugating and Converting, and then Lateral Corrugating. The web page includes a picture of the lateral corrugator, a schematic of how lateral corrugating works, a graph showing the strength enhancement benefits of lateral corrugating compared to conventional corrugating, and a description of the project.

Status:

Progress is again being made on the construction of the lateral corrugator glue machines. These machines are essential to achieve the next project milestone. With progress made this past quarter and that projected for next quarter, meeting the targets outlined in the Milestones Status Table are expected.

During this past quarter, Pamarco delivered the two applicator rolls and two metering rolls required for the glue machines. Dave Burgess, Executive Vice-President of Sales, coordinated this contribution on behalf of Pamarco. These rolls will be installed in the glue machine frame and mounted to the lateral corrugator.

Also during this quarter, Chicago Electric delivered four gear reducers for the glue machine drive system. Chicago Electric will also provide the drive motors and controllers for the glue machines. Robert Kaska is coordinating activities for Chicago Electric. The motors and controllers should be delivered to IPST within the next few weeks.

Harper Machinery has declined to build the glue machine bodies as promised and has terminated its involvement in the project. IPST will build the glue machine bodies incorporating the components provided by Pamarco and Chicago Electric. This will incur additional costs to IPST and funding to support these activities is being sought.

Plans for Next Quarter:

The goal for the upcoming quarter is to complete the glue machines. Plans for mounting the machines on the lateral corrugator and electrically connecting all drive components will be started in this quarter and installation of the glue machines will begin later this quarter or early the following quarter. Once the glue machines are completed and installed, and the machine drive and hydraulic systems connected, the lateral corrugator roll stack configuration will be tested.

Tri-Star Packaging representatives will visit IPST next Quarter to view the lateral corrugator as part of planning activities associated with the incorporation of a lateral corrugator with facilities presently being constructed in Kentucky. Tri-Star Packaging is planning to install a lateral corrugator in a bulk box plant. Lateral corrugating would be utilized into the production of a tri-wall corrugated structure to generate greater strength per unit weight. The integration of the lateral corrugator into a bulk box line is perhaps the best first application of the technology since corrugating speeds for bulk containers falls at the lower end of the production scale, and heavier weight material generates a disproportionately greater strength improvement over lighter weight materials. Steve Baughman is the owner of Tri-Star and is continuing discussions to incorporate the lateral corrugator in to the bulk line within the next 2 years.

Publications, Presentation and Site Visits:

I attended the Forest Products Subprogram biannual Peer Review in Atlanta, GA from April 4-6th, 2006. A PowerPoint presentation and poster were presented. Among others in attendance at the session involving the lateral corrugator was Steve Baughman, president of Tri-Star Packaging, who is the project commercialization partner.

The lateral corrugator was one program highlighted in the DOE's Energy Efficiency and Renewable Energy Publication Technology Solutions: Public-Private Partnerships Transforming Industry, May 15, 2006.

Joe Springer, DOE, toured IPST's Industrial Engineering Center and Lateral Corrugating facilities on April 19. 2006.

The first quarter '06 progress report to DOE was distributed to selected IPST member companies and project commercial partners.

Milestone Status Table:

ID Number	Task / Milestone Description	Planned Completion	Actual Completion	Comments
1	Heat Transfer Experiments	June 2004	June 2004	
2	Splicer and Seam Analysis	June 2004	June 2004	
3	Address Economic and Production Considerations	Sept 2004	Sept 2004	
4	Identify Commercialization Partner	Dec 2004		Ongoing
5	Design Lateral Corrugator	July 2005	July 2005	
6	Build Lateral Corrugator			
6a	Roll Stack	Dec 2005	Dec 2005	
6b	Drive System and hydraulics	Jan 2006	Jan 2006	Design, fabrication and installation complete; electrical connection to be completed
6c	Glue Machines	Oct 2006		Design completed, glue rolls and gear reducers received
7	Demonstrate Lateral Corrugator (i.e., test roll stack configuration)	Nov 2006		
8	Design Sheeter and Splicer	Feb 2007		
9	Build and Integrate Sheeter/Splicer	Sept 2007		
10	Conduct Testing and Demonstrate Overall Concept	Dec 2007		
11	Final Report	Dec 2007		

Budget Data:

Project Spending and Estimate of Future Spending							
Quarter	**From**	**To**	**Estimated Federal Share of Outlays***	**Actual Federal Share of Outlays**	**Estimated Recipient Share of Outlays***	**Actual Recipient Share of Outlays**	**Cumulative**
	Start	6/30/03		24,521		142,869	**167,390**
3Q03	7/1/03	9/30/03		21,907		10,072	**31,979**
4Q03	10/1/03	12/31/03		14,093		3,104	**17,197**
1Q04	1/1/04	3/31/04		50,183		9,099	**59,282**
2Q04	4/1/04	6/30/04		80,454		103,110	**183,564**
3Q04	7/1/04	9/30/04		64,136		0	**64,136**
4Q04	10/1/04	12/31/04		88,733		36,522	**125,255**
1Q05	1/1/05	3/31/05		87,553		18,623	**106,176**
2Q05	4/1/05	6/30/05		57,620		9,732	**67,352**
3Q05	7/1/05	9/30/05		6,400		8,421	**14,821**
4Q05	10/1/05	12/31/05		16,259		8,421	**24,680**
1Q06	1/1/06	3/31/06		0		8,590	**8,590**
2Q06	4/1/06	6/30/06		0		6,493	**6,493**
3Q06	7/1/06	9/30/06	35,000		32,097		**67,097**
4Q06	10/1/06	12/31/06	35,000		40,000		**75,000**
1Q07	1/1/07	3/31/07	25,000		35,000		**60,000**
2Q07	4/1/07	6/30/07	20,000		35,000		**55,000**
3Q07	7/1/07	9/30/07	10,079		30,000		**40,079**
4Q07	10/1/07	12/31/07	0		27,702		**27,702**
Totals			**125,079**	**511,859**	**199,799**	**365,056**	**1,201,793**

Acoustic Forming for Enhanced Dewatering and Formation

Aidun: IPST at Georgia Tech

ID14267

As of August 15, 2006, the PI has not yet submitted an updated status report for the quarter ending June 2006.

Fibrous Fillers to Manufacture Ultra High Ash / Performance Paper

Mathur: G.R. International, LLNL

ID14439

As of August 15, 2006, the PI has not yet submitted an updated status report for the quarter ending June 2006.

Contactless Real-Time Monitoring of Paper Mechanical Behavior During Papermaking

Lafond: IPST at Georgia Tech, LBNL

ID14344

QUARTERLY PROGRESS REPORT

Project Title: Laser Ultrasonics Web Stiffness Sensor

Covering Period: April 1, 2006 to June 30, 2006

Date of Report: July 31, 2006

Recipient: Institute of Paper Science and Technology at Georgia Tech, Atlanta, GA

Award Number: DE-FC07-02ID14344

Subcontractors: none

Other Partners: ABB Industrial systems (Industrial partner)

Contact(s): Emmanuel Lafond, Tel.: 404-894-3707 emmanuel.lafond@ipst.gatech.edu

Project Team: DOE Program Manager: Isaac Chen; Research Team: IPST@GT: Emmanuel Lafond, Gary Baum, David Huggins, Xinya Zhang, collaborating at LBNL: Paul Ridgway

Project Objective: The objective is to provide a sensor that uses non-contact, laser ultrasonics to inspect the mechanical state of paper during the manufacturing process. Tasks include optimization of ultrasound generation on moving paper, development of interferometric detection schemes for on-line operation, and construction of a prototype for single point application on a paper machine.

Background: Laser ultrasonic methods have the potential to greatly extend the utility of on-line ultrasonic telemetry. Existing on-line ultrasonic techniques using contact transducers function only on board grades. Laser ultrasonic methods could perform at higher speeds without causing damage to lightweight papers. Laser ultrasonic methods are able to determine the bending stiffness of the paper. Bending stiffness is a property that is currently measured off-line on paper that determines end-product rigidity and is of great importance in a wide variety of paper grades. Laser ultrasonics could also provide single-sensor in-plane and out-of-plane characterization and give the first on-line gauge of stiffness orientation.

This project is the continuation of project DE-FC07-97ID13578. It is the combined efforts of two organizations with complementary experiences in paper physics and laser ultrasonics. Lawerence Berkeley National Laboratory (LNBL) is expert in the art of laser acoustic wave generation. Progress here is necessary to induce the largest possible ultrasonic

disturbance without damaging the sheet. They developed a scanning-mirror, Mach-Zehnder interferometer that works well at high speeds on a web-simulator. IPST at GeorgiaTech contributes paper physics expertise and close relations with the paper industry as well as laser expertise. They have also demonstrated laser ultrasonic capabilities by constructing unique laboratory ultrasonic systems for use on paper. The group continues to work together to improve these technologies for web measurements. ABB is our industrial partner and is contributing web stablization technology, sensors enclosures, sensor design advices, and softwares.

Status:
This quarter we obtained a verbal agreement from the NorthWest Energy Alliance to support part of our long term alpha plus trial.

In April 2006 IPST and LBNL had a conference call involving several project managers and sponsors of the NorthWest Energy Efficiency Alliance (NEEA) located in Portland, Oregon regarding the stiffness sensor. What was proposed to NEEA was a 6 month trial of the sensor at the Boise St. Helens, OR mill which would be sufficient to demonstrate significant savings in fibers and energy on the paper machine #4 of the mill.

The schedule of the trial is as follows: about 6 months for the design, construction, and testing of the sensor at IPST and LBNL, and 6 months of running the sensor on the machine in St. Helens. The people working on the project on the LBNL side will be Paul Ridgway with a bit of Rick Russo's time. At IPST at Georgia Tech, Emmanuel Lafond, Gary Baum and David Huggins will be working on this 6 months installation. As we did in the past, we will divide the tasks between IPST and LBNL and work together to assemble and test the sensor. But contrarily to the Jackson, AL mill installation, the sensor will be independent from the ABB head package and will be a single CD position sensor. It will be installed close to the ABB platform, on the dry end of the machine. The CD position however will be changeable by the operator to allow the operator to measure the stiffness in the center, and the edges of the web.

There will be some redesign compared to the Jackson trial sensor to allow easier maintenance, repair and servicing of the sensor while the web and ABB Smart Platform are running. At this stage a sensor independent from the ABB head package is needed so as not to interfere with daily operations of the machine during 6 months. A program of the various tasks was drafted and approved in May-June after modifications following feedback from NEEA. As NEEA and the Dept. of Energy have convergent goals - increasing energy efficiency, and decreasing energy consumption of paper machines per ton of paper produced -, we think this is an exciting opportunity to conclude successfully this DOE project with the support of NEEA.

In April through June, Emmanuel Lafond and David Huggins, the electrical engineer who was hired to replace Ted Jackson, worked on reconnecting the sensor from the Jackson trial to the ABB Smart platform and on understanding and documenting the procedures for the operation of the sensor and of its software. This is very important as the St. Helens sensor will reuse most of the communication software with the ABB platform, from the Jackson sensor.

This quarter David Huggins was working only 20% of the time on the project as we are not yet in the design & build stage for the long term sensor. Early on we discovered that the ABB Smart platform of IPST had some serious issues related to a power outage several months ago. After many long hours and great troubleshooting support and advice from Ake Hellstrom and other ABB personnel, we were able to restore first the basis weight and caliper sensor to full operation, to get the head package to standardize properly, and finally in June, to re-establish communication with the service workstation (computer monitoring ABB's sensor) and with the laser ultrasonics sensor. Once more, ABB's help and dedication proved invaluable to restart this Smart Platform.

At the end of June we still have a few communications issues between the sensor and ABB platform to troubleshoot. These deal with the tapping of information of basis weight and caliper from the Smart platform and we are working on them. Funding from NEEA is expected anytime now, once there is agreement on intellectual property between IPST and LBNL. The kick-off meeting of the NEEA project and DOE long term installation is planned for July 28 at the mill in St. Helens.

Plan for next quarter:

- Finalize and sign NEEA contracts both at IPST and LBNL
- Hold kick-off meeting for long-term (Alpha plus) trial at Boise
- Design and build sensor hardware for extended Alpha Plus trial in close relationship with ABB and Boise

Tentative budget from July 1, 2006 till December 31, 2006 for IPST at Georgia Tech:

3rd calendar quarter 2006: $40 k
4th calendar quarter 2006: $50 k

Development of METHANE deNO$_X$ Reburning Process for Wastewood, Sludge, and Biomass Fired Stoker Boilers

Bryan: Gas Technology Institute

GO10418

QUARTERLY PROGRESS REPORT

Project Titles: Development of METHANE de-NOX® Reburning Process for Wood Waste and Biomass Fired Stoker Boilers

and

Utilization of Non-Condensable Gases as Reburn Fuel in FPI Wood Waste and Sludge-Fired Stoker Boilers

and

Advanced METHANE de-NOX for Woodwaste-Fired Stoker Boilers

Covering Period: April 1 to June 30 2006

Date of Report: July 31, 2006

Recipient: Gas Technology Institute (GTI)
1700 Mount Prospect Road
Des Plaines, IL 60018-1804

Award Number: DE-FC36-99GO10418

Subcontractors: ESA Environmental Solutions, Sargent & Lundy LLC, Fluent Engineering, Reaction Engineering and University of Illinois at Chicago

Other Partners: Gas Research Institute, IGT Sustaining Membership Program, Detroit Stoker Company, Boise Cascade and Georgia-Pacific Corporation

Contact(s):

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Project Team: Gibson Asuquo, DOE project manager; Carrie Capps, DOE Project Monitor; David Highsmith, Georgia-Pacific Port Hudson Operations Project Manager; Tom Gilmore, Boise DeRidder mill project manager; Chad Stodola, Boise International Falls project manager; Larry Szymanski, ESA Environmental Solutions; Tom Giaier, Detroit Stoker Company project manager; Fluent Inc., Reaction Engineering Inc, University of Illinois at Chicago.

Project Objective: The primary project objective is to demonstrate the effectiveness of the METHANE de-NOX® (MdN) process for promoting more efficient use of wood waste and sludge for steam generation while keeping NO_x and CO emissions in compliance for biomass–fired stoker boilers in the Forest Products Industry (FPI). A second objective is to investigate the use of non-condensable gas (NCG) and stripper off gas (SOG) as reburn fuel in the MdN technology. This process extension enhances existing reburn technology benefits by further reducing natural gas consumption while providing an effective waste gas disposal option.

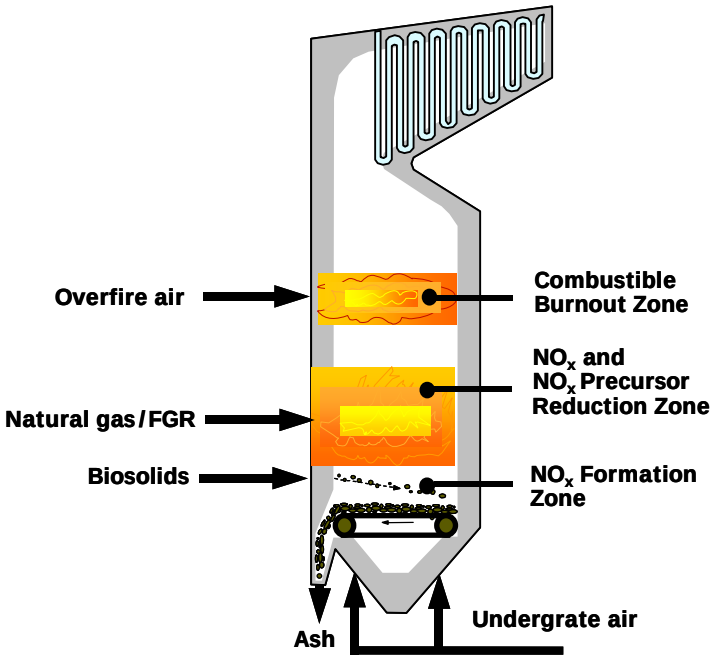


Figure 1. MdN reburn process uses fuel and air staging for combustion improvement and reduced NOx emissions

Background: The firing of biosolids can be limited by the low heating value and presence of bound nitrogen in these fuels. High moisture fuels result in inefficient combustion due to the latent heat of water vapor that is lost to the stack. High fuel moisture biosolids can also contribute to poor fuel distribution and piling, resulting in poor undergrate air distribution, uneven combustion at the grate, and increased emissions of CO and NO_x. Fuels with high nitrogen content such as secondary and tertiary treatment solids also contribute to increased NO_x emissions, limiting the ability to fire these fuels in boilers operating near their NO_x permit limit. Cofiring supplemental fuel such as natural gas through auxiliary burners helps to improve combustion effectiveness and to reduce NO_x emissions. However, these benefits are typically limited to the fractional input of the cofiring fuel.

The MdN reburn process uses both fuel- and air-staging to improve combustion and reduce boiler emissions. A small amount of natural gas and recirculated flue gas (FGR) is injected above the stoker grate to create a well-mixed, oxygen-deficient atmosphere immediately above the primary combustion zone (Figure 1). Air distribution between the undergrate and overfire air is also adjusted to reduce oxygen availability in the lower furnace and improve burnout in the upper furnace. Hotter and less-oxidizing conditions at the grate promote the decomposition of fuel-bound nitrogen compounds to reduce nitrogen oxide formation, while deeper air staging improves burnout in the upper furnace and allows operation with lower excess air. The added heat release and gas mixing at the stoker grate also improves combustion of difficult-to-burn woodwaste fuels. As a result, more high-moisture waste fuels can be burned, while reducing NO_x emissions, stabilizing combustion and improving boiler efficiency through reduced carbon losses and operation with lower excess air.

An MdN boiler retrofit consists of four primary components: 1) a natural gas supply and injection system, 2) a flue gas recirculation system, 3) air distribution adjustments that may include

overfire air system modifications, and 4) control integration. Depending on many operational conditions and constraints, which vary widely for FPI woodwaste-fired boilers, the MdN process can reduce NO_x emissions by 30 –50% and improve boiler thermal efficiency up to 2%, while stabilizing grate combustion and increasing the ability to fire difficult-to-burn fuels.

The project has resulted in the successful demonstration of the MdN technology on a bark- and sludge-fired boiler at a Boise Cascade paper mill in International Falls, MN and baseline testing and evaluation of two additional bark-fired stoker boilers at other mills. As a result of this testing, a continuation project was awarded in 2001 under Cooperative Agreement DE-FC36-99GO10418 to extend the application and benefits of the technology to include a flexible combustion-based disposal option for NCG through their use as reburn fuel in bark and hog fuel boilers. In this project, an existing NCG collection and distribution system will be incorporated into a reburn system retrofit to be developed and demonstrated on a 200,000 lb/h MCR (maximum continuous rating) bark-fired boiler at a second Boise mill in DeRidder, Louisiana.

Another MdN system installed at a Georgia- Pacific paper mill at Port Hudson, LA in 2002 achieved NO_x reduction of over 30% on a 225,000-lb/h bark and gas-cofired boiler. A second continuation project awarded in May 2004 has resulted in the design and installation of a modified overfire air (OFA) and flue gas recirculation (FGR) system to further improve the boiler's energy and emissions performance.

Quarter Status for Base and Continuation Projects:
During this quarter, a second draft review of the project final report covering proprietary considerations of the submitted material was completed and the report finalized. The project final report consists of four commercial design packages showing the MdN reburn technology applied to four (4) different types of wood waste-fired stoker boilers:

1. Stoker boiler with a square furnace, traveling grate, and low volume high concentration NCG injected through furnace rear wall
2. Stoker boiler with a rectangular furnace, stationary pinhole grate, and no NCG
3. Stoker boiler with a rectangular furnace, traveling rotograte, and high volume low concentration NCG injected under grate.
4. Stoker boiler with a square furnace, vibrating hydrograte (water-cooled) and low volume high concentration NCG injected through furnace rear wall

The final report submittal is planned to concur with the end date of the contract, 12/31/2006.

GTI continues collaboration ESA (the technology licensee) for promoting the technology in industrial applications. ESA is currently evaluating Boise's I. Falls No. 2 Boiler OFA performance, which included development of a CFD model. An MdN OFA system retrofit is under consideration for the No. 2 Boiler.

In another initiative, ESA is evaluating an MdN OFA system retrofit on a stoker boiler at Boise's pulp and paper operations in DeRidder LA. At present, no further information is known..

GTI is also pursing interest in evaluating the use of highly reactive solids as an alternative reburn fuel instead of natural gas. Options under consideration include mill waste streams such as dried sludge or sanderdust. Currently, GTI has had discussions with a major forest products company interested in such a scheme. The company has an installed industrial stoker boiler operating in a plywood manufacturing mill located in Cleveland, Texas. The stoker boiler is constructed with a stationary grate and fuel consists of wood wastes including sanderdust. GTI is in communication with the boiler manufacturer and is gathering details about the boiler's operation and construction in order to make a preliminary retrofit assessment.

Base Project: Development of METHANE de-NOX® Reburning Process for Wood Waste and Biomass Fired Stoker Boilers

This development effort was divided into two phases. In Phase 1, a 300-MMBtu/h wood waste and sludge-fired stoker boiler at Boise Cascade's paper mill in International Falls, MN was retrofitted with MdN technology. Site testing was conducted in December 1999. Field results from 15 parametric tests proved the technology's effectiveness by meeting all projected performance goals as follows:

- Increased sludge firing by over 150% from the current 1.2-1.5 tph to 4 ton per hour
- Increased thermal efficiency for 40-100% load by 1 to 2%
- Reduced NO_x emissions by over 50% compared to previous cofiring mode
- Decreased natural gas input by 25% compared to the previous cofiring mode

Boise Cascade accepted the MdN retrofit and American Forest and Paper Association (AF&PA) recognized MdN's performance benefits with a 1999 Environmental and Energy Achievement Award in the Energy and Management and Innovation Category. The I. Falls system has logged over 4 years of continuous operation without a major problem reported since being placed into continuous full-scale operation on December 12, 1999.

Phase 2 activities in the base project consisted of a variety of tasks to promote the technology transfer and use of MdN within the Forest Products Industry. Please refer to the Milestone Status Table given later in the report for a task description.

Work Summary

Phase 1, Tasks 1 – 3
All work is complete.

Phase 2, Tasks 4 – 10
All work is complete.

Phase 2, Task 11 MdN Technology Database for Wood Firing
Task suspended and moved into the MdN Reburn Technology Manual work per the Modified SOW.

Phase 2, Task 12 MdN Engineering Design
Task suspended and moved into the MdN Reburn Technology Manual work per the Modified SOW.

Phase 2, Task 13 Commercialization and Technology Transfer
All work is complete. Commercialization activities have continued through collaboration with ESA, the technology licensee.

Continuation Project 1: Utilization of Non-Condensable Gases as Reburn Fuel in FPI Woodwaste and Sludge-Fired Stoker Boilers (MdN with NCGs)

The continuation project seeks to use waste gases produced during the pulp and paper process to enhance benefits already demonstrated by the base MdN process through a modification scheme that effectively processes these wastes to further improve energy and environmental performance from base MdN technology.

Background

Discussions with mill personnel at Boise's DeRidder Mill in Louisiana raised the possibility of using the mill's low volume high concentration (LVHC) and stripper off gases (SOG) as a portion of the reburn fuel for the system. This approach can reduce the use of expensive cofiring fuels such as oil and natural gas beyond the 25-30% reduction demonstrated with the conventional base reburn system at International Falls. By introducing LVHC and SOG in the primary combustion zone above the grate (Figure 2), the system should meet MACT control option requirements while reducing or eliminating the need for firing the auxiliary gas burners. NCG use has the potential to further increase gas savings from the reburn system due to substitution of NCG for a portion of the reburn gas (natural gas). In addition, if adequate and reliable destruction of NCG is demonstrated in the system, it will be possible to shut off the gas burners in one or both of the mill's bark-fired boilers when not needed for steam production.

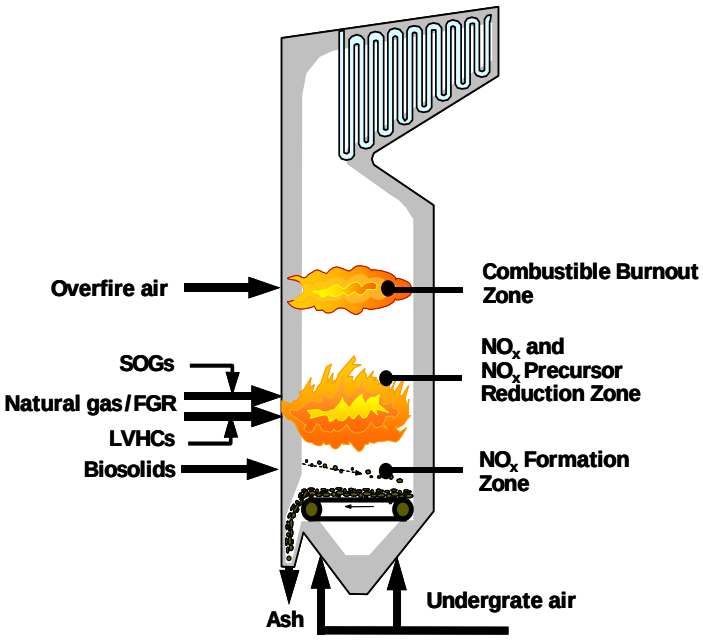


Figure 2. SOG and LVHC will be introduced separately into the natural gas/FGR injection headers

MdN with NCGs performance objectives are:

- utilize to the maximum extent possible all low-volume high-concentration (LVHC) NCG and SOG waste gases consistent with safe and reliable operation
- decrease reburn natural gas consumption by more than 25% compared with original MdN operation
- reduce NO_x emission by over 40% compared to baseline (uncontrolled) conditions
- improve carbon burnout and increase boiler efficiency.

As noted in last quarter's report, the DeRidder mill is under new ownership and the planned MdN retrofit installation at the mill's No 2 Power Boiler is on hold. Subsequent discussions between GTI and DOE recognized the unlikelihood of proceeding with a field retrofit. Therefore a contractual modification to the statement of work and a Revised Statement of Objectives (SOO) was finalized with DOE in September 2005. The SOO reallocated planned project work and shifted funds to provide for preparation of an MdN Reburn Technology Manual (Manual) in lieu of the DeRidder MdN with NCG boiler retrofit.

Work Summary

Task 1 Baseline Testing
All work is complete.

Task 2 LVHC and HVLC Gas System Evaluation
All work is complete.

Task 3 Modeling and Conceptual Design
All work is complete.

Task 4 Detailed Engineering
Task suspended, effort reallocated in the development of an MdN Technology Manual.

Task 5 Procurement and Installation
Task suspended, effort reallocated in the development of an MdN Technology Manual.

Task 6 Parametric Testing
Task suspended, effort reallocated in the development of an MdN Technology Manual.

Task 7 Long-Term Testing
Task suspended, effort reallocated in the development of an MdN Technology Manual.

Task 8 Data Processing and Analysis
All work is complete.

Task 9 Model Validation
All work is complete.

Task 10 Project Management
Management of project activities and budget continued.

Continuation Project 2: Advanced METHANE de-NOX for Woodwaste-Fired Stoker Boilers

Task 1 Port Hudson Modeling and Conceptual Design
All work is complete.

Task 2 Port Hudson Detailed Engineering and Installation
All work is complete.

Task 3 Port Hudson Boiler Performance Testing
All work is complete.

Task 4 Port Hudson Data Processing and Evaluation
All work is complete.

Task 5 DeRidder Modeling and Conceptual Design
Task suspended, effort reallocated in the development of an MdN Technology Manual.

Revised Statement of Objectives (SOO)
Change in work scope; contractual modification executed in September 2005 Revised Statement of Objectives given in the SOO Table on page 11.

Modified Statement of Work:

Task 1-17 International Falls
This task covers preparation of already available documentation and already developed design data for a commercial design package of the MdN retrofit which was successfully completed on the Hog Fuel Stoker Boiler located at the International Falls Paper Mill in Minnesota. This stoker boiler consists of a square furnace, pneumatic bark feed distributors, vibrating hydrograte (water-cooled) and low volume high concentration NCG injection.

All work is complete.

Task 29 DeRidder Modeling, Design and Optimization
This task covers the preparation of documentation and development of a commercial design package for a potential MdN retrofit planned for the No. 2 Power Boiler at the DeRidder Paper Mill in Louisiana. This stoker boiler consists of a square furnace; pneumatic bark feed distributors, a traveling grate, and low volume high concentration NCG and stripper off-gas (SOG) injection.

All work is complete.
Task 25 Port Hudson Modeling, Design and Optimization
This task covers the preparation of already available documentation and already developed design data for a commercial design package for the No. 1 Power Boiler at the Port Hudson Paper Mill in Louisiana. In addition, CFD modeling will study the impact on emissions performance with OFA mixed with FGR. This stoker boiler consists of a rectangular furnace; pneumatic bark feed distributors, a traveling rotograte, and high volume low concentration NCG stream injection.

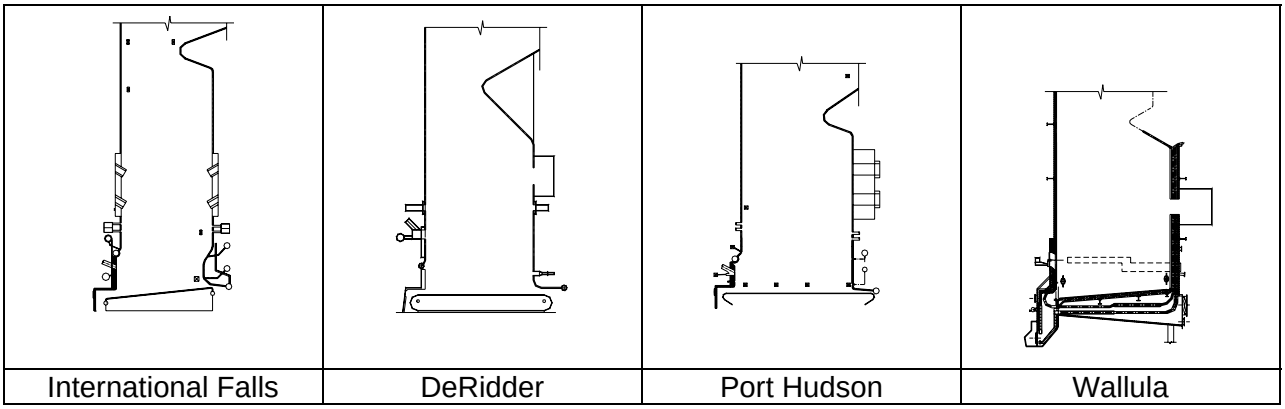
All work is complete.

Task 31 Wallula Modeling, Design and Optimization
This task is new and covers the preparation of documentation and development of a commercial design package for a potential MdN retrofit planned for the Hog Fuel Boiler at the Wallula Paper Mill in Washington. This stoker boiler consists of a rectangular furnace, pneumatic bark feed distributors a stationary pinhole grate and side OFA.

All work is complete.

The MdN Reburn Technology Manual was reviewed for a second time this quarter in order to eliminate any conflicts relating to proprietary nature of report material.

This Manual contains four commercial design packages for the boiler designs illustrated in Figure 3. Each of these boilers's were baseline tested during the course project work. With this manual, potential users can review and understand how MdN technology is adapted; the manual provides retrofit design protocols, CFD baseline and design simulations; retrofit engineering guidelines, retrofit design basics and commercial design specifications.



International Falls	DeRidder	Port Hudson	Wallula

Figure 3 Section views of Stoker Boilers having a Commercial Design Package prepared

Plans for Next Quarter:

1. Continue collaboration as needed with ESA in commercialization initiatives of MdN technology
2. Continue study of alternative reburn fuel options, specifically use of highly reactive solids and solicit letters of interest from industrial clients for DOE funding consideration in 2007.

Patents: None.

Publications/Presentations: None.

Milestone Status Table:

ID Number	Task / Milestone Description	Planned Completion	Actual Completion	Comments
1.0	**MdN at International Falls**			
1.1	Engineering Design for IF Unit #2	9/98	9/98	
1.2	Procurement/Installation at IF Unit #2	11/98	11/99[1]	
1.3	Field Parametric Testing of IF Unit #2	12/98	12/99[1]	
1.4	Long-term Performance Testing at IF Unit #2	7/00	8/00	
1.5	Pilot-Scale Testing at EPA	10/00	--	Task Suspended
1.6	Furnace Computer Modeling	01/01	02/01	
1.7	Boiler 2 Baseline Testing	08/00	04/01	
1.8	Boiler 3 Baseline Testing	08/00	11/01	
1.9	IF Unit #2 Simulation	12/00	10/01	
1.10	Data Processing and Analysis	02/01	12/01	
1.11	MdN Technology Database for Wood Firing	02/01		Task Suspended
1.12	MdN Engineering Design	02/01		Task Suspended
1.13	Commercialization and Technology Transfer	03/01	12/02	
1.14	Project Management and Reporting	04/01		See 3.6
2.0	**MdN with NCGs at DeRidder**			
2.1	Baseline Testing	04/02	06/02	
2.2	LVHC and HVLC Gas System Evaluation	05/02	03/03	
2.3	Modeling and Conceptual Design	08/02	06/03	
2.4	Detailed Engineering	12/02		Task Suspended
2.5	Procurement and Installation	04/03		Task Suspended
2.6	Parametric Testing	06/03		Task Suspended
2.7	Long-Term Testing	09/03		Task Suspended
2.8	Data Processing and Analysis	09/03	5/05	
2.9	Model Validation	11/03	4/05	
2.10	Project Management	09/03		See 3.6
3.0	**Advanced MdN at Port Hudson & DeRidder**			
3.1	Port Hudson Modeling and Conceptual Design	05/04	05/04	
3.2	Port Hudson Detailed Engineering and Installation	05/04	05/04	
3.3	Port Hudson Boiler Performance Testing	08/04	08/04	
3.4	Port Hudson Data Processing and Evaluation	08/04	08/04	
3.5	DeRidder Modeling and Conceptual Design Review	09/04	09/04	New Date 04/05[3]
3.6	Project Management	09/04		New Date 12/05[3]
	Final Report	03/31/05		New Date 12/31/06

Revised Statement of Objectives Status Table

Task Number	Task / Milestone Description	Planned Completion	Actual Completion	Comments
1-17	**MdN at International Falls** Commercial MdN Design Package	12/06		New Date 12/31/06
29	**DeRidder Modeling, Conceptual Design and Optimization** CFD Optimization Study Conceptual Design Commercial Design Package	12/06		New Date 12/31/06
25	**Port Hudson Modeling, Conceptual Design and Optimization** CFD Optimization Study Conceptual Design Commercial MdN Design Package	12/06		New Date 12/31/06
31	**Wallula Modeling and Conceptual Design** CFD Modeling Study Conceptual Design Commercial MdN Design Package	12/06		New Date 12/31/06

Budget Data

Project Spending and Estimate of Future Spending							
Quarter	**From**	**To**	**Estimated Federal Share of Outlays***	**Actual Federal Share of Outlays**	**Estimated Recipient Share of Outlays***	**Actual Recipient Share of Outlays**	**Cumulative**
	Start	9/30/2004		$ 1,410,810		$ 1,926,856	$ 3,337,666
4Q04	10/1/2004	12/31/2004	$ 26,831	$ 47,003	$ 5,380	$ 46,292	$ 93,295
1Q05	1/1/2005	3/31/2005	$ 50,000	$ 90,332	$ 5,382	$ 17,050	$ 107,382
2Q05	4/1/2005	6/30/2005	$ 100,000	$ 75,842	$ 5,382	$ 0	$ 75,842
3Q05	7/1/2005	9/30/2005	$ 120,000	$ 158,005	$ 5,382	$ 0	$ 158,005
4Q05	10/1/2005	12/31/2005	$ 120,000	$ 24,967	$ 5,382	$ 32,754	$ 57,720
1Q06	1/12006	3/31/2006		$ 11,910		$ 1,059	12,790
2Q06	4/1/2006	6/30/2006		$ 0		$ 0	0
Actual Totals				**$ 1,830,711**		**$ 2,024,011**	**$ 3,854,722**
Contract Totals				**$ 1,829,826**		**$2,024,011**	**$ 3,854,722**

*Update quarterly

Steam Cycle Washer for Unbleached Pulp

Salminen: Port Townsend Paper, INL

GO14304

QUARTERLY PROGRESS REPORT

Project Title: STEAM CYCLE WASHER

Covering Period: 2Q06 (April 1, 2006 to June 30, 2006)

Date of Report: July 31, 2006

Recipient: Port Townsend Paper Corporation

Award Number: DE-FC36-04GO14304

Subcontractors:
21st Century Pulp & Paper, LLC
1329 State Street, Suite 202
Bellingham, WA 98225

Other Partners:
Idaho National Laboratory (INL)
P.O. Box 1625
Idaho Falls, ID 83425-2210

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President
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Project Team:
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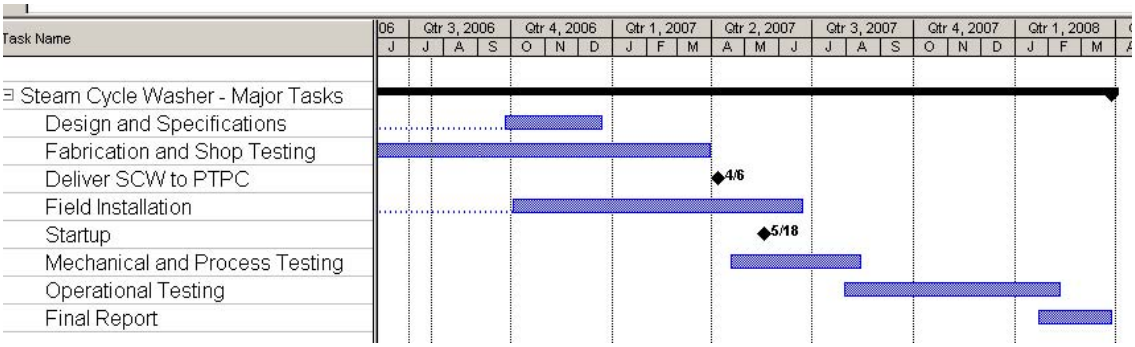
Dean Harding, INL
Advisory Engineer, Industrial and Material Technologies
208-526-6111
dean.harding@inl.gov

Project Objective: This project will provide a commercial scale demonstration of the Steam Cycle Washer (SCW) for Unbleached Pulp. It will show that the SCW will provide an innovative, energy efficient means to wash unbleached pulp using a pressure vessel charged with steam. The benefits of the SCW are that it will enhance mill profitability by significantly reducing energy costs, increasing product quality, and ensure environmental compliance exceeding current regulations.

Background: The feasibility of the SCW concept has been previously demonstrated through extensive pilot plant evaluations. Proprietary, unpublished test results confirm that the SCW will allow pulp mills to substantially reduce energy consumption, exceed environmental compliance requirements, increase fiber quality, and improve washer discharge consistency.

The Port Townsend Paper Corporation's pulp mill in Port Townsend, WA has been selected as the host site for conducting the demonstration of the SCW. This technology can be installed with minimal downtime and production impact to the mill by installing it in parallel to the existing brown stock washers. Startup and initial testing of the SCW will occur in 2007.

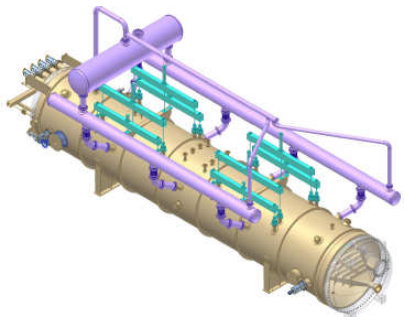
Steam Cycle Washer Major Tasks: (revised 7/21/06)



Status: This is the eighth quarterly report (2Q06) of the SCW project. 2Q06 began with Alaskan Copper, in Seattle, Washington, working to finalize detailed engineering designs, specifications, and biddable shop drawings; Jesse Engineering, of Tacoma, Washington, continuing fabrication of the wire table assembly; and Checkmate Control Systems, Inc, of Issaquah, Washington, continuing design of the process control software and P&Id's. As we moved further into the quarter it became apparent that the organizational and funding issues with 21st Century Pulp & Paper were affecting the ability of the project to effectively move ahead. Consequently the design efforts were suspended while the partners in 21st Century worked through their issues. Fabrication at Jesse Engineering also slowed but machining on several components continued. It is expected that major activities on the project could be further delayed four to eight months. I have modified the schedules in this report to show what I currently believe is a best-case scenario with respect to project timing.

Major Task 1: Design and Specifications for the SCW:

Subtask 1: Design and Specifications: The basic SCW engineering design and specifications has been completed by 21st Century P&P, and Alaskan Copper. During the quarter the work on the preparation of final, approved fabrication drawings by Alaskan Copper was temporarily suspended. Final approved fabrication drawings by Alaskan Copper will be delivered to Jesse Engineering once the project is resumed at the end of 21st Century's arbitration process. Alaskan Copper will then finalize the engineering specifications, stress analysis, and fabrication drawings for the pressure vessel, ancillary piping, and filtrate tanks. Currently the expectations are that the final fabrication drawings will be completed in 4Q06.



SCW External View

Subtask 2: Algorithms and P&Ids Development: Checkmate Control System's work on the development of the SCW process control software programming, and instrumentation design was also suspended during the quarter. There is a possibility that we could permanently lose the services of CCSi because of the delay created while 21st Century resolves their organizational and funding issues. To mitigate this Port Townsend Paper Corporation will divert the some of the DOE grant funds that they are holding to contract directly with CCSi instead of CCSi being a subcontractor to 21st Century. Checkmate (CCSi) will continue to update the redlined P&ID's, work on electrical and control panel design, develop plant-specific electrical tie-in drawings, and start programming of the PLC. Purchase and delivery of the PLC equipment and associated software will be rescheduled to coincide with the shop-testing phase of the project.

Subtask 3: Design Field Piping: Most of the tie-ins required for the SCW were completed during the annual plant shutdown in October 2005. Field piping runs are currently being laid out and installation of field piping should start in 4Q06.

Major Task: 2: Fabrication and shop testing.

Subtask 1: Fabrication: Alaskan Copper in Seattle, Washington was finalizing the engineering specifications and fabrication drawings for the pressure vessel, ancillary piping, and filtrate tanks when their work was suspended. Budget pricing has been solicited and received for the fabrication of the pressure vessel. Completion of the stress analysis of the SCW pressure vessel

and finalization of the fabrication drawings is not expected to resume until 4Q06. At that time a contract will be awarded for completion of the pressure vessel. After completion of fabrication of the SCW pressure vessel it will be delivered to Jesse Engineering for final assembly and shop testing.

Fabrication on the SCW interior components (e.g. wire table, perimeter frame, pressure plate, and wash header assemblies) was started at Jesse Engineering in 4Q05. During 2Q06 fabrication efforts were slowed pending receipt of the final approved fabrication drawings from Alaskan Copper. Currently Jesse Engineering is machining on some of the SCW components using a 2-man crew. Upon completion, these internal components will be staged for the final assembly of the SCW.

Purchased materials staged for fabrication.

Wire table in fabrication.

Subtask 2: Shop Testing: Shop testing of the assembled washer will occur at Jesse engineering before delivery to the Port Townsend Paper mill. Current plans are that completion of SCW fabrication, assembly, shop testing, and required modifications will be completed during 1Q07 or early 2Q07.

Major Task: 3: Delivery and Field Installation

Sub Task 1: Delivery: With the aid of the P&IDs, Port Townsend and 21st Century Pulp & Paper have identified the pumps, motors, valves, instruments, piping, etc for both the new SCW piping and changes to the existing mill systems. Long delivery items such as pumps, motors, and electrical switchgear have been delivered to Port Townsend during 2Q06. Bids have been solicited for installation of the foundations and support structure but a contract has not been awarded. Final purchase orders for unpurchased materials and installation should be written during 3Q06 and 4Q06.

Sub Task 2: Field Installation: Installation of the structural steel piling and footings will start in 4Q06. Erection of the SCW structural steel supports and platform will be completed in 1Q07 in anticipation of the arrival of the SCW at Port Townsend Paper.

Milestone Status Table:

Steam Cycle Washer for Unbleached Pulp				
Quarter	Task/Milestone	Planned Completion	Actual Completion	Comments
3Q04	-Receive notification of Federal Assistance Award -Begin to set up reporting and accounting formats	-9/20/04 -9/20/04	-9/20/04 -9/30/04	
4Q04	-Complete setting up reporting and accounting formats -Conduct organizational meeting to identify roles, responsibilities, and project timing -Begin permitting process for Steam Cycle Washer project -Start detailed shop drawings and specifications -Establish washer location and tie-ins to existing mill infrastructure	-10/29/04 -11/30/04 -11/1/04 -10/15/04 -12/15/04	-12/17/04 -11/1/04 -1/18/05 -12/04 -meetings held on 11/30/04 & 12/28/04	-21st Century will "invoice mill for all its charges -No Ecology Notice of Construction is required -Alaskan Copper Works is preparing drawings -Hipp Engineering will complete installation details
1Q05	-Complete detailed shop drawings (Finish Design) -Start to write algorithms for process automation -Start development of plant-specific Process and Instrumentation Drawings (P&IDs) -Select support equipment (pumps, motors, valves, instruments, piping, etc) to support P&ID requirements	-3/31/05 -1/31/05 -2/17/05 -12/01/05	-9/30/05 -6/30/05 -3/31/05	 -hired Tim Ooyman on from Checkmate Control Systems -use INL and Checkmate for development of P&IDs -budget quotes obtained from several vendors but final selection not complete
2Q05	-Complete algorithm development -Complete hydraulic system design and specifications -Complete P&IDs -Start to develop mill-specific drawings for piping and foundation fabrication	-6/30/05 -6/10/05 -6/24/05 -5/16/05	9/30/05 -2Q06 -5/16/05	 -Incorporating redline changes into P&ID's -Foundation drawing complete and piping drawings are in progress
3Q05	-Bid, evaluate, and award contracts for fabrication and installation -Start fabrication of field piping and washer foundations -All permitting requirements complete -Start installation of foundations, tie-ins, piping, and E&I	-7/12/05 -8/1/05 -9/16/05 -9/19/05	 -1/24/06 -10/3/05	-not yet awarded; will carry into 3Q06 - Most fieldwork will be restarted in 4Q06. -building permits were approved in 1Q06 - piping tie-ins complete; will start foundations in 4Q06
4Q05	-Assemble and test the Steam Cycle Washer and its associated equipment at fabrication shop (Complete Fabrication) -Complete installation of field piping, tie-ins, and foundations -Write memorandum regarding finished shop test results -Start installation of Steam Cycle	-11/21/05 -12/30/05 -12/16/05 -12/26/05		-Expect this to be delayed to early 2Q07 (4/6/07) -will carry over to 2Q07 -can't be completed until 2/Q/07 -delayed to 2Q07

	Washer at mill			
Steam Cycle Washer for Unbleached Pulp				
Quarter	Task/Milestone	Planned Completion	Actual Completion	Comments
1Q06	-Complete field installation of Steam Cycle Washer including E&I -Train pulp mill personnel on the operation of Steam Cycle Washer -Conduct mechanical and process-technical trial runs -Start development and testing of optimum operating parameters for the Steam Cycle Washer and batch digesters -Write memorandums regarding mechanical trial runs, process- technical trial runs, and commissioning and acceptance of Washer into mill operations	-1/20/06 -2/4/06 -2/25/06 -2/28/06 -3/31/06		-will carry into 1Q07 -expect to occur during 2Q07 -2Q07 and 3Q07 -3Q07 -2Q07
2Q06	-Continue development and testing of optimum operating parameters and batch digester recipes for all the common wood species (fir, hemlock, and cedar) -Begin selection and testing of the optimum operating parameters for the paper machine separately for each paper grade produced by the pulp mill -Write a presentation paper regarding the operation of the Steam Cycle Washer using only data that 21st Century Pulp & Paper does not consider being proprietary. -Write a presentation paper regarding unbleached pulp and paper characteristics after processing with the Steam Cycle Washer	-Ongoing -4/4/06 -4/15/06 -6/30/06		-4Q07 and 1Q08 -4Q07 -4Q07 -1Q08
3Q06 & 4Q06	-Continue development and testing of optimum operating parameters and batch digester recipes for all the common wood species (fir, hemlock, and cedar) -Continue selection and testing of the optimum operating parameters for the paper machine separately for each paper grade produced by the pulp mill -Continue to write presentation papers regarding unbleached pulp and paper characteristics after processing with the Steam Cycle Washer -Complete Field Validation	-Ongoing -Ongoing 12/31/06		-3Q07 thru 1Q08 -1Q08 -1Q08 -2Q08

Plans for Next Quarter:

During 3Q06 21st Century Pulp and Paper efforts will be directed at resolving their organizational and funding issues. Refinement of the SCW P&ID's by Checkmate Control Systems will resume. Jesse Engineering will proceed with fabrication and machining of the SCW internal components using the drawings that they have received to date. Efforts of Alaskan Copper on final design and completion of fabrication drawings will continue on hold. Port Townsend will continue the purchase of equipment required for the field installation of the SCW; continue with the design of the piping runs connecting the completed tie-ins with the SCW; and contract with an installation contractor. Port Townsend Paper expects to start installation of the washer support structure foundations in early 4Q06. INL's efforts in drafting performance auditing test procedures will also continue with minimal activity through 4Q06.

Patents: None

Publications/Presentations: None

Budget Data as of 7/25/06

Port Townsend Paper Corporation - 12943								
Steam Cycle Washer for Unbleached Pulp								
Actual Project to Date Spending and Estimate of Future Spending ($) - DOE and Recipient (PTPC)								
Quarter	From	To	Estimated Federal Share of Outlays	Actual Federal Share of Outlays	Invoices Received for Federal Share of Outlays	Estimated Recipient Share of Outlays	Actual Recipient Share of Outlays	Cumulative
3Q04	7/1/04	9/30/04	109,064	109,064	0	0	0	109,064
4Q04	10/1/04	12/31/04	100,000	100,000	79,234	47,550	47,550	256,614
1Q05	1/1/05	3/31/05	1,050,000	1,050,000	139,813	49,117	49,117	1,355,731
2Q05	4/1/05	6/30/05	0	0	436,971	42,576	42,576	1,398,307
3Q05	7/1/05	9/30/05	232,746	232,746	714,546	204,467	204,467	1,835,520
4Q05	10/1/05	12/31/05	0	0	219,000	402,464	402,464	2,237,984
1Q06	1/1/06	3/31/06	278,190	278,190	0	94,970	94,970	2,611,144
2Q06	4/1/06	6/30/06	0	0	0	162,356	162,356	2,773,500
3Q06	7/1/06	9/30/06	0	0		50,000		2,823,500
4Q06	10/1/06	12/31/06	0	0		1,100,000		3,923,500
1Q07	1/1/07	3/31/07	0	0		1,600,000		5,523,500
2Q07	4/1/07	6/30/07	0	0		750,000		6,273,500
3Q07	7/1/07	9/30/07	0	0		275,000		6,548,500
4Q08	10/1/07	12/31/07	0	0		63,000		6,611,500
1Q08	1/1/08	3/31/08	0	0		134,500		6,746,000
Totals			1,770,000	1,770,000	1,589,564	4,976,000	1,003,500	6,746,000

= estimate

Port Townsend Paper Corporation - 12943					
Steam Cycle Washer for Unbleached Pulp					
Actual Project to Date Spending and Estimate of Future Spending ($) - DOE Laboratory Partner (INL)					
Quarter	From	To	Estimated DOE Lab Share of Outlays	Actual DOE Lab Share of Outlays	Cumulative
3Q04	7/1/04	9/30/04	0	0	0
4Q04	10/1/04	12/31/04	0	0	0
1Q05	1/1/05	3/31/05	37,435	37,435	37,435
2Q05	4/1/05	6/30/05	66,340	66,340	103,775
3Q05	7/1/05	9/30/05	29,346	29,364	133,121
4Q05	10/1/05	12/31/05	43,460	43,460	176,581
1Q06	1/1/06	3/31/06	62,500	62,500	239,081
2Q06	4/1/06	6/30/06	27,926	27,926	267,007
3Q06	7/1/06	9/30/06	10,000		277,007
4Q06	10/1/06	12/31/06	10,000		287,007
1Q07	1/1/07	3/31/07	15,000		302,007
2Q07	4/1/07	6/30/07	15,000		317,007
3Q07	7/1/07	9/30/07	15,000		332,007
4Q08	10/1/07	12/31/07	15,000		347,007
1Q08	1/1/08	3/31/08	52,993		400,000
Totals			400,000	267,025	400,000

Port Townsend Paper Corporation - 12943								
Steam Cycle Washer for Unbleached Pulp								
Actual Project to Date Spending and Estimate of Future Spending ($) - Recipient								
Quarter	From	To	Current Estimated 21st Century Share of Outlays	Actual 21st Century Share of Outlays	Current Estimated Port Townsend Share of Outlays	Actual Port Townsend Share of Outlays	Total Recipient Share of Outlays	Cumulative
3Q04	7/1/04	9/30/04	0	0	0	0	0	
4Q04	10/1/04	12/31/04	43,260	43,260	4,290	4,290	47,550	47,550
1Q05	1/1/05	3/31/05	20,770	20,770	28,347	28,347	49,117	96,667
2Q05	4/1/05	6/30/05	27,210	27,210	15,366	15,366	42,576	139,243
3Q05	7/1/05	9/30/05	46,575	46,575	157,892	157,892	204,467	343,710
4Q05	10/1/05	12/31/05	146,300	146,300	256,164	256,164	402,464	746,174
1Q06	1/1/06	3/31/06	50,000	50,000	44,970	44,970	94,970	841,144
2Q06	4/1/06	6/30/06	55,000	55,000	107,356	107,356	162,356	1,003,500
3Q06	7/1/06	9/30/06	0		50,000		50,000	1,053,500
4Q06	10/1/06	12/31/06	500,000		600,000		1,100,000	2,153,500
1Q07	1/1/07	3/31/07	1,000,000		600,000		1,600,000	3,753,500
2Q07	4/1/07	6/30/07	350,000		400,000		750,000	4,503,500
3Q07	7/1/07	9/30/07	150,000		125,000		275,000	4,778,500
4Q07	10/1/07	12/31/07	45,000		18,000		63,000	4,841,500
1Q08	1/1/08	3/31/08	115,885		18,615		134,500	4,976,000
Totals			2,550,000	389,115	2,426,000	614,385	4,976,000	4,976,000

= estimate under review

Demonstration of Black Liquor Gasification at Big Island

DeCarrera: Georgia-Pacific Corporation

NT40850

Report 40850R22

Quarterly Technical Progress Report 22
Demonstration of Black Liquor Gasification at Big Island

Reporting Period Start Date: **April 01, 2006**
Reporting Period End Date: **June 30, 2006**

Principal Author: **Robert DeCarrera**

Reporting Date: **July 25, 2006**

DOE Award Number: **DE-FC22-01NT40850**

Submitted By: **Georgia-Pacific Corporation**
133 Peachtree Street, N.E.
Atlanta, Georgia 30303

DISCLAIMER

ABSTRACT

This Technical Progress Report provides an account of the status of the project for the demonstration of Black Liquor Gasification at Georgia-Pacific Corporation's Big Island, VA facility. The report also includes budget information and a milestone schedule.

Additional information may also be found on the project web site listed below:
http://www.gp.com/containerboard/mills/big/steam.html

TABLE OF CONTENTS

Note: Any section marked by an asterisk is required (by DOE) in all technical reports.

SUBCONTRACTORS

Since the project has moved from the construction phase to the commissioning phase, the list of subcontractors has been removed from the report.

PROJECT DESCRIPTION

The project to be conducted by G-P is a comprehensive, complete commercial-scale demonstration that is divided into two phases. Phase I was the validation of the project scope and cost estimate. Phase II is project execution, data acquisition and reporting, and consists of procurement of major equipment, construction and start-up of the new system. Phase II also includes operation of the system for a period of time to demonstrate the safe operation and full integration of the energy and chemical recovery systems in a commercial environment.

The objective of Phase I was to validate the process design and to engineer viable solutions to any technology gaps. This phase included engineering and planning for the integration of the full-scale MTCI/StoneChem PulseEnhanced™ black liquor steam-reformer chemical recovery system into G-P's operating pulp and paper mill at Big Island, Virginia. During this phase, the scope and cost estimate was finalized to confirm the cost of the project including integration into the existing system at the mill.

The objective of Phase II of the project is the successful and safe completion of the engineering, construction and functional operation of the fully integrated full-scale steam reformer process system. This phase includes installation of all associated support systems and equipment required for the enhanced recovery of both energy and chemicals from all of the black liquor generated from the pulping process at the Big Island Mill. The objective also includes operation of the steam reformer system to demonstrate the ability of the system to operate reliably and achieve designed levels of energy and chemical recovery while maintaining environmental emissions at or below the limits set by the environmental permits.

SUMMARY

The project continued Phase II commissioning and liquor runs during this reporting period. Liquor run 20 on Reformer 2 started 4/30/06 with liquor injection starting 5/02/06. The run was still in progress at the end of the period. Liquor run 21 was started 5/15/06 with liquor injection starting 5/17/06. Liquor run 21 was terminated on 6/26 as part of the mill's flood preparation plan.

The total cost of the project through April 2007 is currently estimated to be $156.8 million. This reflects no change from the previous forecast. Project-to-date spending through June 2006 is $132.4 million. The Defense Contract Audit Agency (DCAA) audit of CY 2001 costs was issued in April 2006 with a qualified opinion. A significant amount of our claimed subcontractor costs relating to the prime contractor, Fluor were not audited due to their failure to submit an incurred cost submission in a format acceptable to DCAA. DCAA has classified the Fluor costs as unresolved pending the audit of their costs and will issue a supplemental report upon incorporation of the requested assist audit. The audit of CY 2002 Georgia-Pacific costs is nearly complete and will be issued by DCAA in August 2006. The audit of CY 2003 Georgia-Pacific costs will begin the week of 7/24/06 and is projected to be complete in September 2006. The audit for CY 2004 has not been scheduled. Georgia-Pacific's CY 2005 incurred costs are being compiled and will be submitted in the August 2006 timeframe.

On 4/6/06, the project team hosted an EPA Project XL meeting at the mill. On 4/11-12/06, the project team participated in the Steam Reformer User's Group meeting held at the Norampac mill in Trenton, Ontario, Canada.

GP has determined the Kraft liquor trial is not feasible with the technology as it is installed in Big Island. During the next period, GP will process the necessary forms to remove this item from the project scope. The pulse heater tubes have experienced carburization. During the Steam Reformer Roundtable II (Sept. 2005), presentations were made by metallurgists from Fluor and Oak Ridge National Labs. Both agreed that our tubes would remain serviceable for several years at our current bed and liquor conditions. Both also agreed that an increase in liquor sulfur concentration (Kraft liquor) would expose the pulse heaters tubes to problems which could be potentially severe. This includes accelerated carburization as well as a risk of polythionic stress corrosion cracking of the tubes following the exposure to higher sulfur liquor. The cost to replace the tubes in the pulse heaters is approximately $250,000 per heater. The MTCI unit trialed at New Bern on Kraft liquor experienced polythionic SCC failure on 253MA alloy tubes. Although 253MA is more susceptible to this than our 321SS alloy, performing a Kraft liquor trial poses a risk that is not justified.

Another reason this trial is not feasible is due to the significant amount of tar contained in the reformer product gas. GP does not have any data on the projected composition of tars from the Kraft liquor or their impact on plugging of the gas clean up system. There is no feasible method of determining this composition or quantifying the impact on the gas clean up system.

SAFETY and ENVIRONMENTAL

There were no recordable incidents during this reporting period.

We currently stand at six OSHA recordable incidents on the project:

- 04/24/01 - see Quarterly Technical Progress Report 02
- 09/18/02 - see Quarterly Technical Progress Report 07
- 01/09/04 - see Quarterly Technical Progress Report 13
- 02/13/04 - see Quarterly Technical Progress Report 13
- 03/25/04 - see Quarterly Technical Progress Report 13
- 09/07/05 - see Quarterly Technical Progress Report 19

There were no environmental incidents during the reporting period. We currently stand at 2 environmental incidents (see Quarterly Technical Progress Report 04) thus far on the project.

All of the project environmental reporting and permits are current.

ACCOMPLISHMENTS

Engineering / Project Management / Procurement / Construction

Engineering is supporting commissioning activities by designing changes to the process and equipment as experience is gained during the liquor runs.

During this period, the project team:

- Continued reformer trials to assess potential changes to reduce tar formation and improve carbon conversion.
- Conducted trials with modified tips for the liquor injectors to measure the impact on tar formation and carbon conversion.
- Replaced failed sections of the Reformer 1 pulse heater exhaust ducts.
- Installed a rental cooling tower to allow the process to run while additional process engineering was performed to assess the cooling needs for the Gas Cleanup system.
- Purchased and installed individual flow meters on liquor injectors for Reformer 1.
- Completed GP's support of the DOE funded portion of the process research at the University of Utah.
- Began pilot trials to serve as the design basis for a tar removal and collection system.
- Installed air distributors in the pulse heater air plenums to improve combustion air distribution.
- Completed trials to determine pulse heater performance with fewer gas jets on the aerovalves.
- Installed long pulse heater combustion chamber thermocouples in Reformer 2.
- Replaced all of the refractory in the combustion chambers of Reformer 2 with Harbison-Walker Greencast 94Plus.
- Successfully performed trials cooling the reformer bed with water as part of the shut down process.
- Evaluated the performance of the sootblower in HRSG 1 and determined it was successful.
- Purchased a new green liquor filter for installation in early 2007.
- Continued to research alternatives to eliminate the tar formation and low carbon conversion issues.
- Participated in the April Project XL meeting at Big Island.

- Participated in the Steam Reformer User's Group meeting held at the Norampac mill in Trenton, Ontario, Canada.

Training

During this period, operator training was limited to new operational procedures which were updated as experience was gained during the trials.

Commissioning

Liquor run 20 on Reformer 2 was started on 4/30/06 with liquor injection on 5/02/06. This run was still in progress at the end of the period. During this run, several failures of the fabric expansion joints on the pulse heater exhaust ducts were experienced. As previously reported, sections of this duct between the reformer and the expansion joints were replaced with stainless steel duct due to the failure of the internal lining of the original duct. The design of the duct by the supplier did not properly account for the new thermal expansion of the stainless duct. The increased thermal expansion of the stainless duct caused a displacement in the expansion joints which eventually led to failures. Also during this run, failures of the internal lining of the vertical sections of this duct were discovered. Temporary repairs were implemented to the expansion joints and fans were used to cool the vertical sections. The design of replacement ducts and expansion joints was completed.

Liquor run 21 on Reformer 1 was started on 5/15/06 with liquor injection on 5/17/06. This run was terminated on 6/26/06 as part of the mill's flood preparation plan. Due to the length of time required to cool the bed and remove it to the Media Storage Silo, the shut down decisions for the reformers occur very early in the flood preparation plan. Shutdown of Reformer 2 was also scheduled to follow, but the prediction for the river crest was lowered and the unit was kept on line. During this run, Reformer 1 also experienced failures of the pulse heater expansion joints and vertical duct. After run 21, 4 water leaks were discovered in the tube sheet of PH1-1. These leaks were weld repaired and the tubes were plugged. Also, a crack in the weld between the reformer vessel and the PH1-2 Decoupler was discovered. This crack allowed bed solids to be blown from the reformer to the exhaust duct of the pulse heater. Inspection of the other units revealed similar cracks. All of these were weld repaired.

During the period when both reformers were in service, the heat load on the Cooling Tower was above the operating capabilities of the tower. Modifications made to the cooling system in an attempt to allow operation of both units at full load were not successful. On 6/12/06, a rental cooling tower was placed in service to eliminate this problem. Engineering data continued to be collected at the end of the period to better size a replacement tower.

For the shutdown of run 19, the project team successfully used water injection to cool the reformer bed. For the start up of run 21, the project team successfully started the unit up with only 3 pulse heaters covered with bed solids. Liquor injection was used to increase the bed level to the normal operating level before PH1-4 was placed on main flame. By accomplishing both of these items, the scope of the Dry Bed Removal system will be significantly reduced.

ACTIVITIES NEXT QUARTER

- Continue reformer trials to assess problems and increase reliability.
- Investigate and trial modified tips for liquor injectors and changes in liquor process variables to measure the impact on tar formation and carbon conversion.
- Replace vertical sections of the Reformer 1 pulse heater exhaust ducts.
- Replace vertical sections of the Reformer 2 pulse heater exhaust ducts.
- Begin installation of new aerovalve assemblies as available.
- Install sootblower in HRSG 2.
- Design system and purchase additional cooling tower equipment to address the problem of insufficient cooling tower capacity.
- Analyze the cause of tube sheet leaks in pulse heaters and work to develop remedy.
- Continue to support process research at the University of Utah and complete trials for liquor injector design.
- Perform pilot trials to serve as the design basis for a tar removal and collection system.
- Continue evaluation of metallurgy issues on the pulse heater tubes and tube shields.

PROJECT ISSUES AND CONCERNS

During the liquor runs we continue to work on issues and concerns. Some of these issues are specific only to this project, while others apply to the technology.

- Tar Formation – This remains a major project issue at this time. The testing conducted has shown that tar formation will continue to be an issue. A chemical dispersant has proven to help reduce the plugging of the liquid circuits, but has not been effective in the vapor piping. GP will continue pilot trials to serve as the basis for the design of a tar separation and removal system.
- Carbon Conversion – Neither GP nor Norampac have been able to achieve the predicted carbon conversion. GP's conversion rate is approximately 60% as compared to an expected 98.4%. GP has begun engineering for the installation of a higher capacity green liquor filter to reclaim the carbon from the bed solids for fuel or other uses. Installation of the new green liquor filter is expected in early 2007. In spite of the excess carbon, the filtered green liquor quality produced by the reformers has been very good.
- Liquor Injection System – The liquor injectors were modified to a flat spray design. GP will continue to work in this area and collect data to evaluate process changes. Procedures to allow for unplugging of injectors while the reformer is in service have eliminated some of the previous issues.
- Aerovalve Design –A revised design for the aerovalve plate assembly was tested during this period. The design changes were successful in reducing the operating temperature of the valves. Additional units have been ordered and will be installed later this year.
- Pulse Heater Integrity –The Pulse Heater tubes and tube shields have experienced carburization. Metallurgists report that the pulse heater service life may not be affected by this problem. GP has installed tubing of various materials for longer term testing in service. Inspections after the last few outages of Reformer 1 have identified several tubes which have bent. The actual cause has not been identified. After liquor run 21, 4 leaks were located in the PH1-1 tube sheet. At least 2 of these leaks were associated with tubes that were deformed. Additional engineering and research will be required to determine the root cause of the tube deformation and the root cause of the tube sheet leaks.

- Pulse Heater Heat Transfer – Preliminary data indicates that the pulse heaters are only accomplishing 60% to 70% of the designed heat transfer to the bed. Additional data will be taken on future runs.
- Bed Particle Size –Bed particle size has a significant effect on fluidization, carbon conversion, heat transfer from the Pulse Heater and bed motion. The effectiveness of controlling particle size by steam attrition and/or by varying liquor injection parameters will require additional operational experience to evaluate. During runs 20 and 21, bed particle size has remained within the operating range at all liquor injection rates without the need for steam attrition.
- Pulse Heater Combustion Chamber Refractory – The Express 30 installed after run 15 failed during run 17. Analysis of the failures indicated the operating temperature was higher than the design rating of the refractory. The analysis estimated the temperatures were in the range of 2800 ^{o}F to 3200 ^{o}F. For the next runs, a longer thermocouple was installed in a pulse heater to correlate the temperature of the center of the combustion chamber with the original measurements near the outside edges of the chamber. New operating parameters were used to control the temperature in the chambers to less than 2600 ^{o}F. As reported last period, GP installed several different refractory for testing. All of the refractory materials installed performed well.

PROJECT COSTS

Budget Data

Project Budget Period	Period			Cumulative		
From - To	DOE	G-P	Total	DOE	G-P	Total
04/02/06-06/30/06	$0	$ 3,835,416	$3,835,416	$44,239,118	$88,195,207	$132,434,325
Previous Total	$44,239,118	$84,359,791	$128,598,909	33.4%	66.6%	100.0%

Note: Table has been modified to reflect actual payments by DOE.
DOE retains $200,000 pending final report.

DOE Obligated Funds			
Amendment	Amount	Total	Date
000	$4,537,776	$4,537,776	2/13/2001
001	$0	$4,537,776	4/25/2001
002	$0	$4,537,776	5/3/2001
003	$0	$4,537,776	8/13/2001
004	$0	$4,537,776	8/17/2001
005	$4,671,000	$9,208,776	9/10/2001
006	$13,130,000	$22,338,776	9/17/2001
007	$162,665	$22,501,441	9/26/2001
008	$0	$22,501,441	10/31/2001
009	$0	$22,501,441	11/6/2001
010	$6,385,000	$28,886,441	2/1/2002
011	$3,684,000	$32,570,441	9/16/2002
012	$44,540	$32,614,981	9/30/2002
013	$4,000,000	$36,614,981	3/24/2003
014	$4,408,000	$41,022,981	5/7/2003
015	$3,000,000	$44,022,981	4/5/2004
016	$416,141	$44,439,122	5/11/2004
017	$0	$44,439,122	5/27/2004
018	$0	$44,439,122	7/26/2004
019	$0	$44,439,122	10/20/2004
020	$0	$44,439,122	3/28/2005

SCHEDULE

The Project Milestone Summary has been deleted from the report. The only activities planned from this point forward are to continue liquor runs on both reformers to gain operating experience and improve system reliability. GP will announce the final decision on the acceptance of the technology on or before 3/01/07.

EXPERIMENTAL

There were no experimental items conducted during this period.

GP is partially funding a study at the University of Utah to evaluate a liquor injector design. Results of this study will be available later this year.

The project team has provided support to various DOE supported R&D projects during previous periods. Currently DOE is not funding any R&D projects in support of this technology.

RESULTS and DISCUSSION

This report is for a demonstration project that is conducting liquor runs. The performance data continues to indicate the process is significantly below the target levels for product gas production due to low carbon conversion and the formation of tars and soot. The primary focus of the project has shifted to improve the reliability and operability of the process. As a secondary focus, liquor runs and research efforts will continue to stress improvement of the performance of the units by increasing carbon conversion and reducing the production of tars. Alternate uses for the carbon and tars are also being investigated.

CONCLUSION

This report is for a demonstration project that is not yet completed. As such, conclusions listed below are based on trials conducted to date.

- As liquor flow is increased to a reformer, the majority of this incremental fuel value will convert to increased bed carbon and increased tar and soot in the product gas.
- The typical RPG heating values are in the range 280 – 315 BTU /scf compared to an expected 260 BTU/dscf.
- Internally generated RPG can only supply approximately 75 – 85 % of the required pulse heater fuel input.
- Process must be modified to capture tar and soot from the product gas as well as residual carbon in the bed solids to become net energy positive.
- Demonstrated carbon conversion to RPG at full load is 60% compared to an expected 98.4%.
- RPG production at full load for one reformer is currently 1800 dscfm compared to an expected 4600 dscfm.
- Generally the ratio of reformer product gas (RPG) to natural gas (NG) is approximately 2.8 compared to an expected 5.0. The measured values have a slight trend upward at higher liquor loads.

REFERENCES

All of the reference reports used in the preparation of this report are in a preliminary form. Once the reports are finalized the information will be included.

Development of Screenable Wax Coatings and Water-Based Pressure Sensitive Adhesives

Severtson: University of Minnesota

GO14309

DOE F 4600.3A
(03-94)

U.S. Department of Energy

OMB Control No.
1910-0400

Milestone Log

OMB Burden Disclosure Statement

Program/Project Title:	Program/Project ID No.:
Development of Screenable Wax Coatings and Water-Based Pressure Sensitive Adhesives	DE-FC36-04GO14309

ID No.	Description	Planned Completion Date	Actual Completion Date	Comments As of (Date)
1	Characterization and removal testing of Franklin label grade water-based PSAs.	10/05	10/05	Completed as scheduled
2	Characterization and removal testing of standard wax coatings.	10/05	10/05	Completed as scheduled
3	Characterization and removal testing of new model water-based PSAs. Go/No-Go Point ***Criterion for proceeding*** **– Properties identified as those governing fragmentation are confirmed**	10/06		• Model systems have been tested and the results confirm our hypotheses. Further modifications have been made and these new PSAs will be tested this summer to confirm results.
4	Study on the role of facestock properties in determining removal of PSAs.	10/06		• PSA laminates produced with Franklin PSA formulations and a variety of facestocks were sent to FPL for testing using a new laboratory protocol.
5	Characterization and removal testing of new model wax coatings. Go/No-Go Point ***Criterion for proceeding*** **– Properties identified as those governing removal are confirmed**	04/07		• New model wax coatings are being generated using novel nanoparticles. These will be characterized and their performance properties measured.
6	Development of new benign commercial PS labels. Go/No-Go Point **Criterion for proceeding – Laboratory results confirmed for PS labels at pilot scale**	11/07		• Performance testing is being carried out on model formulations that demonstrated high removal efficiencies in laboratory testing. Plans are already underway to scale-up at least one new formulation.
7	Study on the role of board properties in determining removal of wax coatings.	08/07		
8	Development of new benign commercial treated corrugated containers.	11/07		

Budget Data (07/07/05): The actual spending should reflect the money actually spent on the project in the corresponding periods.

Project Spending and Estimate of Future Spending							
Quarter	**From**	**To**	**Estimated Federal Share of Outlays**	**Actual Federal Share of Outlays**	**Estimated Recipient Share of Outlays**	**Actual Recipient Share of Outlays**	**Cumulative**
4Q04	10/1/04	12/31/04	112,872	94,000	35,853	29,140	123,140
1Q05	1/1/05	3/31/05	104,308	98,494	35,854	34,473	256,107
2Q05	4/1/05	6/30/05	50,942	76,506	35,853	41,600	374,213
3Q05	7/1/05	9/30/05	51,090		35,854	48,201	422,414
4Q05	10/1/05	12/31/05	65,796		29,561	28,361	450,775
1Q06	1/1/06	3/31/06	65,800	225,000	29,560	30,374	706,149
2Q06	4/1/06	6/30/06	65,796		29,561	29,784	735,933
3Q06	7/1/06	9/30/06	65,800		29,560		
4Q06	10/1/06	12/31/06					
1Q07	1/1/07	3/31/07					
2Q07	4/1/07	6/30/07					
3Q07	7/1/07	9/30/07					
4Q07	10/1/07	11/30/07					
Totals			**582,404***	**494,000**	**261,656***	**241,933**	**735,933**

* Values for the first year and one quarter of approved budget.

			Approved Spending Plan			**Actual Spent to Date**		
Phase/Budget Period			DOE Amount	Cost Share	Total	DOE Amount	Cost Share	Total
	From	To						
Year 1	10/04	10/05	319,212	143,414	462,626			
Year 2	10/05	10/06	263,183	118,242	381,424			
Year 3	10/06	10/07	210,731	94,676	305,407			
Totals			793,126	356,332	1,149,458			

QUARTERLY PROGRESS REPORT

Project Title: Development of Screenable Wax Coatings and Water-Based Pressure Sensitive Adhesives

Covering Period: April 1st, 2006, through June 30th, 2006

Date of Report: July 11th, 2006

Recipient: Department of Bio-based Products, University of Minnesota, 2004 Folwell Avenue, St. Paul, MN 55108

Award Number: DE-FC36-04GO14309

Subcontractor: United States Department of Agriculture Forest Service, Forest Products Laboratory

Other Partners: Boise Cascade Corp., Franklin International, The International Group, Inc.

Contact: Steven J. Severtson, (612) 625-5265, sever018@umn.edu

Project Objective: The project objective is the design of new water-based pressure sensitive adhesive (PSA) products and wax coatings that are engineered for enhanced removal during the processing of recycled fiber. Research includes the formulation, characterization, and performance measurements of new screenable PSAs and wax treatments, testing of modified paper and board substrates and the design of test methods to characterize the inhibition of adhesive fragmentation and wax comminution and relative removal efficiencies of developed formulations.

Background: The presence of PSAs and wax coatings in recycled paper creates a number of problems for the recycling process including lost production and diminished product quality. This project will focus on the design of adhesives and coatings that are more effectively removed from the papermaking process during furnish screening. These new materials should possess properties that enhance removal without impacting performance.

Work will include the identification of properties that control adhesive fragmentation and coating comminution and use of this information to design new formulations optimized for both removal and product performance. Through an iterative process where the surface and bulk mechanical properties of materials are characterized and compared against repulping behavior and screening removal efficiencies, the properties controlling fragmentation and comminution will be identified. Products will then be reformulated to manipulate these properties and produce commercially feasible, screenable products. In addition to the development of new adhesives and wax coatings, the role of the paper substrates in determining removal efficiencies will be investigated. Treatments such as strength resins, pigment coatings, sizing agents and others will be used to modify the wet and dry strength of paper, interfacial properties and PSA/coating-paper adhesion. The influence of the modifications on the adhesive fragmentation and coating

comminution will be determined from monitoring the particle size and morphology during repulping operations and by measuring screening removal efficiencies. It is expected that the results of this work will identify combinations of paper and PSA or board and wax treatments that provide for significant screening removal efficiencies of the adhesive or coating layer.

Status: A summary of results obtained during the reporting period by the University of Minnesota and its partners are as follows:

- On April 5th, 2006, S. Severtson (UM), J. Guo (UM), and L. Gwin (Franklin International) participated in the ITP Forest Products Peer Review Meeting 2006 held in Atlanta. A presentation and poster were provided by the P.I. reporting the technical progress, financial status, commercialization potential and advancement through the milestone chart.

- Conference calls were held on April 21st, April 24th, May 19th, June 6th, June 7th and June 26th between L. Gwin (Franklin International), S. Severtson (UM) and J. Guo (UM). These meetings are used to clarify and review information conveyed via emails and phone calls and will often involve other participants from Franklin International and/or the University of Minnesota.

- The initial set of model water-based or emulsion PSAs were synthesized and formulated last quarter. For one system, the surfactant systems used for the emulsification of monomers were juggled amongst various commercial products with removal efficiencies ranging from approximately 0 to nearly 100%. The goal was to determine to what extent emulsifiers influence the fragmentation of PSAs during repulping operations. The results of this study have helped narrow possible contributors to the negative impact PSAs have on recycling operations.

- A second model system focused on the monomer make-up of the adhesive polymer. New emulsions have been synthesized with modifications to the monomer composition using a polymerization method of a Franklin International product which has a laboratory-measured removal efficiency that is less than 5%. Members of the model system demonstrate a broad range of removal efficiencies. The performance and bulk mechanical and surface properties of the new model system are currently being tested. The results have led to the identification of a model system that shows promise for commercialization. Optimization of the monomer composition, surfactant system and coating package is currently underway. Also being examined is the polymerization procedure.

- Three laminates produced with Franklin International PSAs and facestock provided by Boise Cascade were sent to the Forest Products Laboratory. The removal efficiency for these laminates will be tested using a new laboratory-scale test.

- Measurements of the thermal expansion of pure wax and its nanocomposites were conducted using a Perkin-Elmer thermomechanical analyzer (TMA7) equipped with a quartz dilatometer attachment. Preliminary results showed lower linear coefficient of thermal expansions (CTE) for nanocomposites with less than 2 wt% clay concentration compared to

unfilled wax. Wax nanocomposites with lower CTE may find applications that require greater thermal dimension stability.

- Rheological properties of wax and its nanocomposites were further characterized with a TA instruments AR-G2. This unit reportedly is an extremely sensitive rheometer, which is necessary for the unmodified paraffin wax. The effect of clay on the shear rheology of molten wax was investigated. Manuscripts are being written to report the results.

- A conference call was held with representatives of the University of Minnesota and a nanotechnology company. The P.I. will investigate the influence of a novel "nanoparticle," which has reportedly been used in other commercial products to inhibit moisture flow and to enhance mechanical properties. The transfer of samples should begin next quarter. Once obtained, the testing of nanocomposite properties should begin.

- A paper entitled "Significant and Concurrent Enhancement of Stiffness, Strength, and Toughness for Paraffin Wax through Organoclay Addition," was published in *Advanced Materials* (vol. 18, #12, pages 1585-1588). The paper outlines the properties of new organo-clay, wax coating nanocomposites.

Plans for Next Quarter:
The team will focus on Tasks 3, 4 and 5 over the next quarter. Based on the results of testing for model systems, a potentially new commercial product has been identified. Optimization of this formulation will begin next quarter. This will involve optimizing the composition, emulsifier and coating package. The influence of paper facestock will also be examined. If these results are promising, the next step will be the generation of pilot-scale batches. Work will also continue on the characterization and performance testing of wax-based nanocomposites. Several papers are currently being prepared and the influence of a new nanoparticle on the bulk mechanical and surface properties will be examined.

Mechatronic Design and Control of a Waste Paper Sorting System for Efficient Recycling

Venditti: North Carolina State University

ID13880

QUARTERLY PROGRESS REPORT

Project Title: Mechatronic Design and Control of a Waste Paper Sorting System for Efficient Recycling

Covering Period: April 1, 2006 through June 30, 2006, 2nd Quarter 2006

Date of Report: July 19, 2006

Recipient: N. C. State University
Dept. of Wood and Paper Science
Raleigh NC 27695-8005

Award Number: DE-FC36-00ID13880 (switched from DE-FC07-00ID13880)

Subcontractors: None

Other Partners: Advanced Sorting Technology, LLC, 3738 Keystone Ave., Nashville, TN 37211
Weyerhaeuser Co., WTC 2E19, PO Box 9777, Federal Way, WA 98063-9777

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OBJECTIVE

The objective of this research is to develop a sensor network for High Speed Automated Paper type characterization. Past research has led to the development of a high throughput lignin sensor and an experimental concept for a bending stiffness sensor. The possibilities of integrating gloss and color sensors have also been explored. Current work involves the improvement of the robustness of the Stiffness sensor setup and the integration of all the above mentioned sensors into a sorting system. A sorting algorithm to characterize the type of paper based on the data obtained from all the sensors has to be developed.

Introduction

This quarter's work involved the completion of the Sensor Characterization process, the investigation of the flutter phenomenon and the Modification of the Lignin Sensor setup to enable integration with LabVIEW$^{tm.}$ During the previous quarter, we started developing the Paper Material Model so that it may be used in the FEA simulations of the sensor setup. We had established the elastic constants of different paper samples for various orientations with respect to the paper machine direction.

This quarter, we continued by developing a Plasticity Model for the paper sample. However, the plasticity model was not incorporated into the FEA simulations due to computational complexity. We plan to use these results in the future to perform more accurate simulations. Only the elasticity parameters were incorporated into the FEA model for dynamic simulations using ABAQUS. The results of the FEA simulations help us in the characterization of the sensor and also in determining the optimum speed of the conveyor.

Paper Plasticity Model

Most of the plasticity models, which define the relationship between the stress and strain when the material starts deforming plastically, are "incremental" theories. In these the mechanical strain is decomposed into an elastic part and plastic part. In these models, the true strain values are used instead of nominal strain values. This is helpful when the strains are very large such as in a ductile material. True strain, ε also called logarithmic or natural strain, is defined such that every incremental length change is divided by the current length.

$$d\varepsilon = \frac{dl}{l} \quad \xrightarrow{yields} \quad \varepsilon = \int_{l_0}^{l} \frac{dl}{l} \quad \xrightarrow{yields} \quad \varepsilon = ln\frac{l}{l_0}$$

The true strains for equivalent deformation in tension and compression are identical except in sign. The advantage of true strains over the nominal strains is that the true strains are additive, the total strain being equal to the sum of the incremental strains. When the strains are small, then true and engineering strains are nearly equal.

Plasticity models have a yield surface, a flow rule and the evolution laws that define the hardening criteria. Yield criteria is a mathematical expression whose primary use is to predict if or when yielding will occur under combined stress states in terms of particular properties of the material being stressed.

The most general form is $f\left(\sigma_x,\sigma_y,\sigma_z,\tau_{xy},\tau_{yz},\tau_{zx}\right)=C$; *Where, C is a constant*

In terms of Principal stresses, the yield criterion can be expressed as $f(\sigma_1,\sigma_2,\sigma_3)=C$

The flow rule defines the inelastic deformation that occurs if the material point is no longer responding purely elastically and the evolution laws govern the way in which yield and flow definitions change as inelastic deformation occurs. Since the paper is orthotropic in nature, Hill's yield criterion that is used for anisotropic materials is used for defining the plasticity model. Hill's stress function is an extension of the Mises function to model anisotropic behavior.

The function is

$$f(\sigma)=\sqrt{F(\sigma_y-\sigma_z)^2+G(\sigma_z-\sigma_x)^2+H(\sigma_x-\sigma_y)^2+2L\tau_{yz}^2+2L\tau_{yz}^2+2M\tau_{xz}^2+2N\tau_{xy}^2}$$

In terms of rectangular Cartesian stress components, where *F, G, H, L, M, N* are constants obtained by tests of material in different orientations. These constants are defined as

$$F=\frac{(\sigma^0)^2}{2}\left(-\frac{1}{X^2}+\frac{1}{Y^2}+\frac{1}{Z^2}\right)\quad;\quad G=\frac{(\sigma^0)^2}{2}\left(\frac{1}{X^2}-\frac{1}{Y^2}+\frac{1}{Z^2}\right)\quad;\quad H=\frac{(\sigma^0)^2}{2}\left(\frac{1}{X^2}+\frac{1}{Y^2}-\frac{1}{Z^2}\right)$$

$$L=\frac{(\sigma^0)^2}{2}\frac{1}{R^2}\quad;\quad M=\frac{(\sigma^0)^2}{2}\frac{1}{S^2}\quad;\quad N=\frac{(\sigma^0)^2}{2}\frac{1}{T^2}$$

Where σ_o is the reference yield stress and *X, Y, Z* are the tensile yield stresses and *R, S, T* are the yield stresses in shear. Plastic material behavior is predicted using an isotropic hardening formulation. The following is the flow rule that governs the hardening:

$$d\varepsilon_{ij}^{pl}=\frac{\partial f}{\partial\sigma_{ij}}d\lambda$$

Where ε_{ij}^{pl} is the plastic strain; $d\lambda$ is a scalar multiplier that depends on the slope of the hardening curve; $\frac{\partial f}{\partial\sigma_{ij}}$ is the normal to Hill's yield surface. The yield stresses in machine and cross-machine

directions are determined directly from the stress-strain curves. For all properties that could not be obtained from the tests conducted, engineering estimates were made. These were based on the material properties obtained by Baum and Habeger for milk carton stock. The heavy card stock and medium card stock have properties similar to the milk carton stock.

Table 8.3.1. Yield stresses for different paper grades

Yield Stress (Copy Paper)	**Value**	**Anisotropic Yield Stress Ratios**	**Method**
X	25.55 MPa	1	Measured
Y	12.8335 MPa	0.503	Measured
Z	13 MPa	0.509	Estimated
R	13 MPa	0.881	Estimated
S	1 MPa	0.068	Estimated
T	1 MPa	0.068	Estimated
σ^o	25.55 MPa		Estimated
Yield Stress (Medium card stock)	**Value**	**Anisotropic Yield Stress Ratios**	**Method**
X	15.20 MPa	1	Measured
Y	14.22 MPa	0.936	Measured
Z	13 MPa	0.855	Estimated
R	13 MPa	1.481	Estimated
S	1 MPa	0.114	Estimated
T	1 MPa	0.114	Estimated
σ^o	15.20 MPa		Estimated
Yield Stress (Heavy card stock)	**Value**	**Anisotropic Yield Stress Ratios**	**Method**
X	12.79MPa	1	Measured
Y	11.23 MPa	0.878	Measured
Z	13.00 MPa	1.016	Estimated
R	13.00 MPa	1.759	Estimated
S	1.00 MPa	0.135	Estimated
T	1.00 MPa	0.135	Estimated
σ^o	12.79 MPa		Estimated

The Orthotropic elastic and anisotropic plasticity material model is supported only in ABAQUS/Standard analysis code but not in ABAQUS/Explicit. ABAQUS/Standard uses Hilber-Hughes-Taylor operator for integration of the equations of motion. This offers the use of all elements in ABAQUS but can be slower than Explicit. ABAQUS/Explicit uses the central difference operator and is more robust when handling complex contact problems. But Explicit has fewer element types than ABAQUS/Standard. However, the method provided in ABAQUS/Explicit has some important advantages. In explicit problems the analysis cost rises only linearly with the problem size, whereas the cost of solving the nonlinear equations associated with implicit integration rises more rapidly than linearly with problem size.

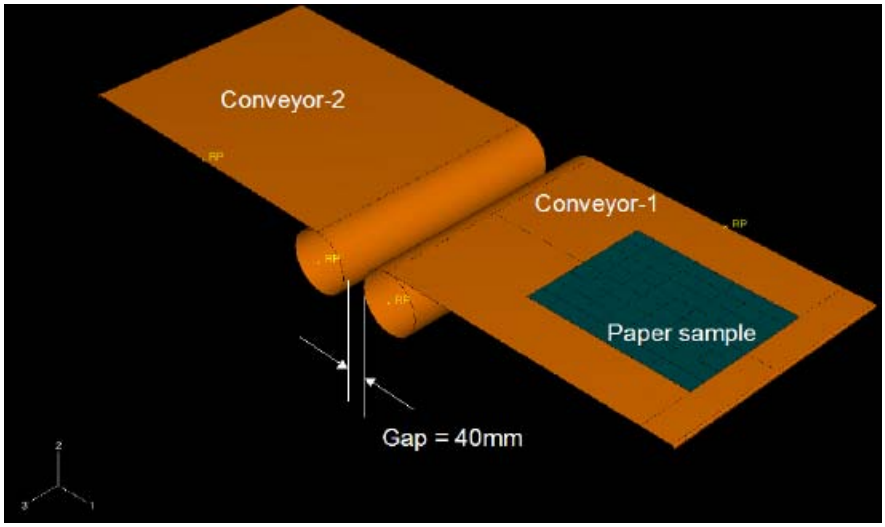
Therefore ABAQUS/Explicit is suitable for simulating the Paper-Conveyor dynamic problem.

FINITE ELEMENT MODEL

In order to find the deflections of the selected paper grades when they are moving on a high-speed conveyor, a finite element analysis model was built. Initially the model was built in ABAQUS/Standard environment. Because of significant nonlinearity involved in the model, the ABAQUS/Standard code was unable to converge. So, the model was built in ABAQUS/Explicit environment, which is suitable for transient dynamic analysis problems.

Model Geometry:

The conveyor surface is modeled as an analytical rigid surface and the paper is modeled as orthotropic lamina. The material constants, which were found using the tensile tester and the error minimization algorithm, are used for building the material model.



Paper sitting on the conveyor surface

Since the lateral (in-plane) dimensions of the paper are much larger than the thickness of the paper, plane stress shell elements are used for modeling the paper surface. These elements also allow transverse shear deformation. Kirchoff's thin shell theory is used for the 3D model, implying that a material line that is originally normal to the mid-surface of the shell elements remains so throughout the deformation. Transverse shear stress acts as penalty function to impose Kirchoff's constraints.

The shell elements that are used for modeling the paper also account for finite membrane strains and arbitrarily large rotations. Each shell element has four nodes at which element variables are calculated. These elements have nodes only at the corners and use linear interpolation in each direction to find the displacements at any other point in the element. Each node has six degrees of freedom these include both the rotational and translational degrees of freedom. Even though the shell elements allow transverse shear deformation, it becomes very small as the shell

thickness decreases. The in-plane dimensions of the paper sample are specified to be equal to 11”x 8.5”.

The machine direction of the paper is specified to be parallel to the length of the paper and cross-machine direction is specified to be parallel to the width of the paper. The machine and cross machine directions of the paper corresponds to the in-plane principal material directions of the paper. The rollers at the end of the conveyor surface are modeled separately as cylindrical analytical rigid surfaces in order to isolate the conveyor surface from the roller surface. This will also allow separate boundary conditions for the rollers. Explicit dynamic analysis procedure is used to solve the nonlinear equations of motion.

Contact Interactions:

Contact interactions are used to model the contact between different surfaces in the model. Contact modeling involves two steps. In the first step various surfaces that might come into contact during the analysis procedure are identified. Then these surfaces are coupled together by specifying them as contact pairs. In the second step mechanical property models are assigned to the contact pairs. The contact property model specifies the normal and tangential behavior of the surfaces when they come into contact.

The normal behavior between the paper surface and the conveyor surface is specified as hard. The “Hard” contact relationship minimizes the penetration of the shell element nodes into the conveyor surface and does not allow transfer of tensile stress across the interface. When the surfaces are in contact, any contact pressure can be transmitted between them. The surfaces separate if the contact pressure reduces to zero. Separated surfaces come into contact when the clearance between them reduces to zero.

When the surfaces are in contact they usually transmit shear as well as normal forces across their interface. The relationship between these two force components is expressed in terms of stresses at the interface of the bodies. This relationship is known as the friction between the contacting bodies. Classical isotropic Coulomb friction model is used to specify the tangential behavior of the contacting surfaces. The standard Coulomb friction model assumes that no relative motion occurs if the equivalent frictional stress is less than the critical stress, τ_{crit} which is proportional to the contact pressure, *p*, in the form

$$\tau_{crit} = \mu p$$

Where μ is the friction coefficient that is defined as a function of the contact pressure *p*. The tangential behavior of the surface is defined by specifying the coefficient of friction between the contacting surfaces. For determining the coefficients of friction between the paper samples and the conveyor, the surface roughness of the conveyor and the paper needs to be known. The

surface properties of the paper samples vary widely during the sorting procedure and it is hard to obtain the coefficients of friction for all possible surface combinations.

Since it is difficult to measure the coefficients of friction, some simplifying assumptions were made. The friction is assumed to be isotropic and it is also assumed that the coefficient of friction does not depend on the contact pressure and other field variables. The coefficient of friction of driving pulley of steel for industrial conveyor is found to be equal to 0.15 from the specifications of the conveyor. The surface roughness of the paper surface will be much less than the surface roughness of the conveyor roller. Therefore the static coefficient of friction between the conveyor and the paper surface is assumed to be equal to 0.07 and the kinetic friction coefficient was set to be equal to 0.04. The effective friction coefficient between the contacting surfaces is assumed to satisfy the following equation

$$\mu = \mu_k + (\mu_s - \mu_k)e^{-dc*\gamma_{eq}}$$

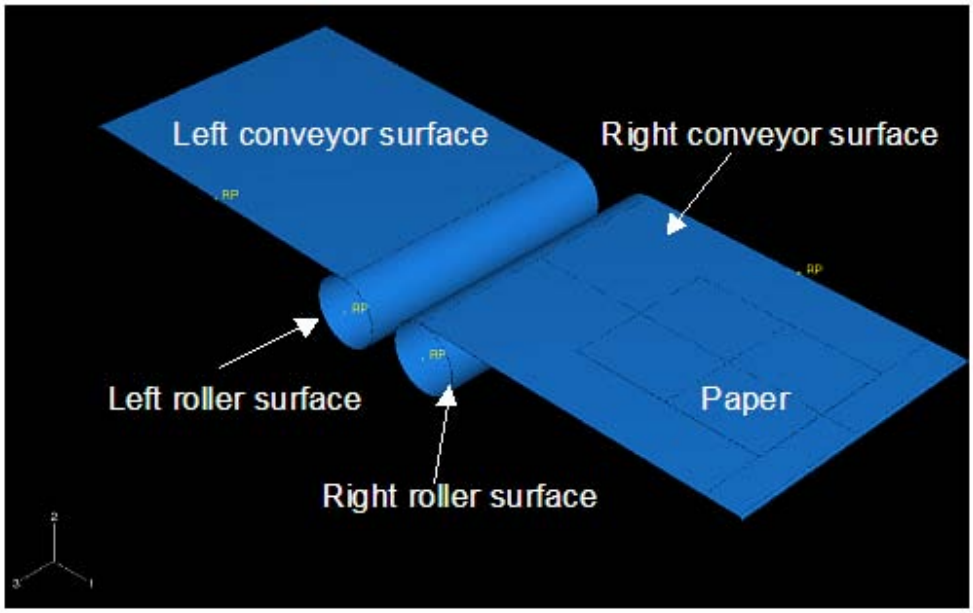
Where, μ_k is the kinetic coefficient of friction, μ_s is the static coefficient of friction, dc is the decay coefficient, γ_{eq} is defined as

$$\gamma_{eq} = \sqrt{\gamma_1^2 + \gamma_2^2}$$

Where γ_1 is the slip rate in direction 1; and γ_{eq} is the magnitude of the slip velocity. The decay coefficient specifies the rate at which the effective friction coefficient value reaches the value of kinetic coefficient of friction once the slip starts to occur.

In the tangential behavior model the decay rate is set to be equal to 0.1. Totally four surfaces are identified in the paper conveyor model that can come into contact with the paper sample during the analysis. Two mechanical contact interaction properties were created. The first property has hard normal behavior and frictionless tangential behavior. For the second property, the tangential behavior is specified to follow the equation for effective friction coefficient. Four contact pairs are created and the interaction properties are assigned to them as shown in the table

Contact Pair	**Interaction Property**	
	Tangential Behavior	**Normal Behavior**
Paper – Right conveyor surface	Frictionless	Hard
Paper – Right roller surface	Friction specified	Hard
Paper – Left roller surface	Friction specified	Hard
Paper – Left conveyor surface	Frictionless	Hard



Potential contact surfaces for the paper sample

The tangential behavior between the paper surface and the conveyor surface is modeled as frictionless because there is no relative motion between the paper and the conveyor surface. Both of them will be moving at the same speed. There will be some slip between the paper surface and the left conveyor surface because the velocity of the paper will be reduced due to the applied pneumatic load. But that slip is neglected because its effect on the paper deflection is insignificant.

Boundary and Loading Conditions:

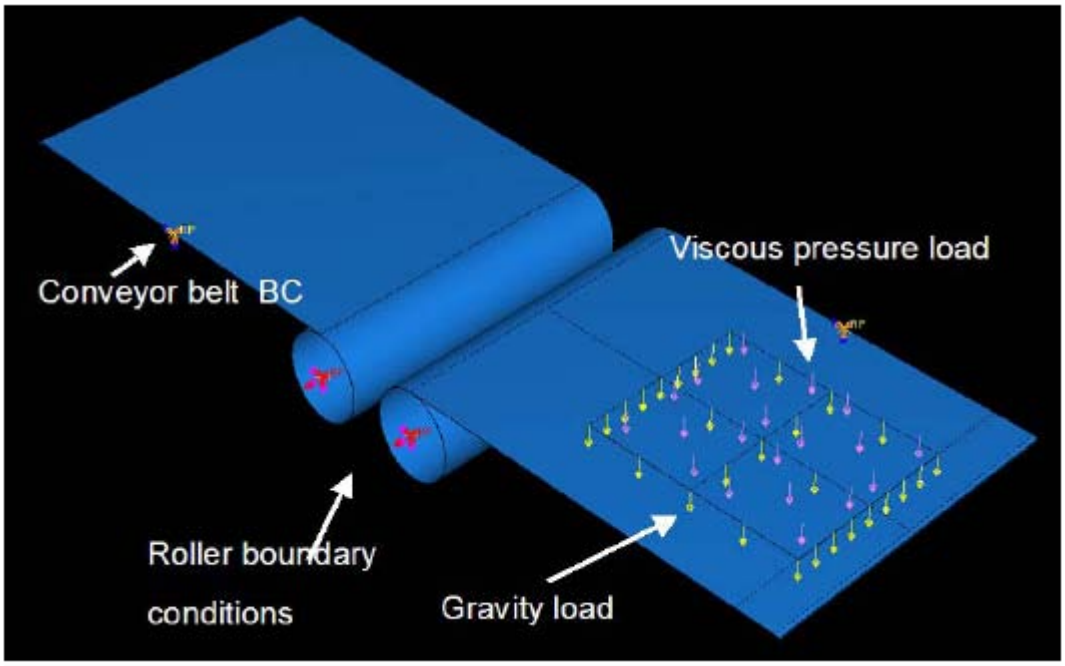
The boundary and the loading conditions specified in the model should be close to the actual system in order to obtain accurate results from the simulation. At the same time, the model should not be made complex by including all the details of the real system. So an optimal method is followed so that the accuracy of the simulation is not compromised and the model is not too complex. The high speed-moving conveyor is modeled by using a flat conveyor surface and a roller. The flat conveyor belt surface is modeled as analytically rigid surface and the roller is modeled as a cylindrical rigid body. The flat conveyor surface is constrained in all directions where as an angular velocity boundary condition is specified for both the conveyor rollers.

To simulate the high-speed moving conveyor, an initial velocity field is specified on the nodes of the paper sample. The initial velocity is made equal to the conveyor speed. But in a real system the paper sample moves along with the conveyor belt and there will not be any relative motion between the two until the paper reaches the end of the conveyor. To meet this requirement, the

tangential behavior between the flat conveyor surface and the paper surface is modeled as frictionless. The external loads acting on the paper sample are load due to surrounding air, weight of the paper and pneumatic load that acts on the paper when it passes over the gap. The load due to the air surrounding the paper sample is modeled as a viscous pressure load whose total load magnitude is given by the expression:

$$p = A \quad P$$

Where p is the total load applied to the paper sample; P is the atmospheric pressure exerted by air.

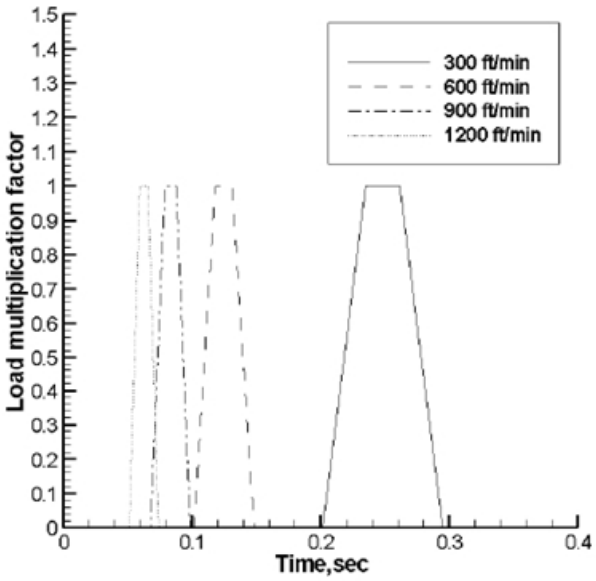


Specific loading and boundary conditions

The pneumatic load applied on the sample can be a uniformly varying load or a concentrated load. Concentrated loading pattern can be obtained by keeping cylindrical jet nozzle close to the conveyor surface. The load intensity and the spread data of the cylindrical nozzle are used for specifying the load on the sample when it passes over the gap.

The load on the sample is applied by first creating a set of elements from the elements on the surface of the paper sample. The element set is obtained by creating a circular partition at the center of the paper surface. The diameter of the circular partition is specified to be equal to the mean spread diameter of the cylindrical nozzle. Once the element set is created, the time history of the load on the element set is prescribed by using the amplitude curves. The amplitude curve specifies the load on a set of elements as a function of time.

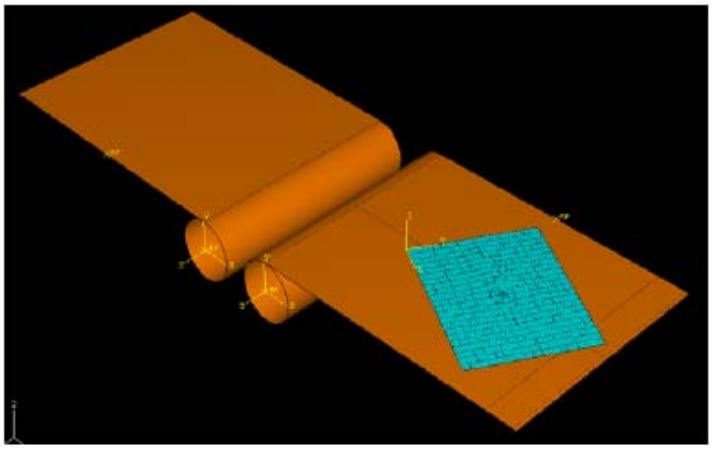
By initially positioning the paper sample at a known distance from the center of the gap, the position of the paper sample as a function of the analysis time can be known from the initial velocity of the paper sample. This information is used to construct the amplitude curves for different conveyor speeds.



Amplitude curves for different conveyor speeds

At any time during the analysis, the actual load applied on the element set will be equal to the product of the maximum load value given on the input data line and the load multiplication factor at that instant of time. The load multiplication factor at any time during the course of the analysis is obtained from the amplitude curve. In this way, the paper samples will be loaded only when they reach the gap between the two conveyors.

The deflection values are obtained for three paper grades as a function of load, conveyor speed and the orientation of the samples on the conveyor belt. Four nozzle loads are used in finding the deflection values of the samples. All the nozzle loads used in the simulation are obtained from the load intensity data of the cylindrical profile nozzle at different inlet pressures when the nozzle is held at a distance of 1" above the conveyor surface. The samples are loaded when the machine direction is oriented parallel to conveyor travel direction, and when machine direction is at 30°, 60°, 90° to the conveyor travel direction. The sample deflection behavior for all the four loading conditions is observed at the conveyor speeds of 300 ft/min and 1200 ft/min.



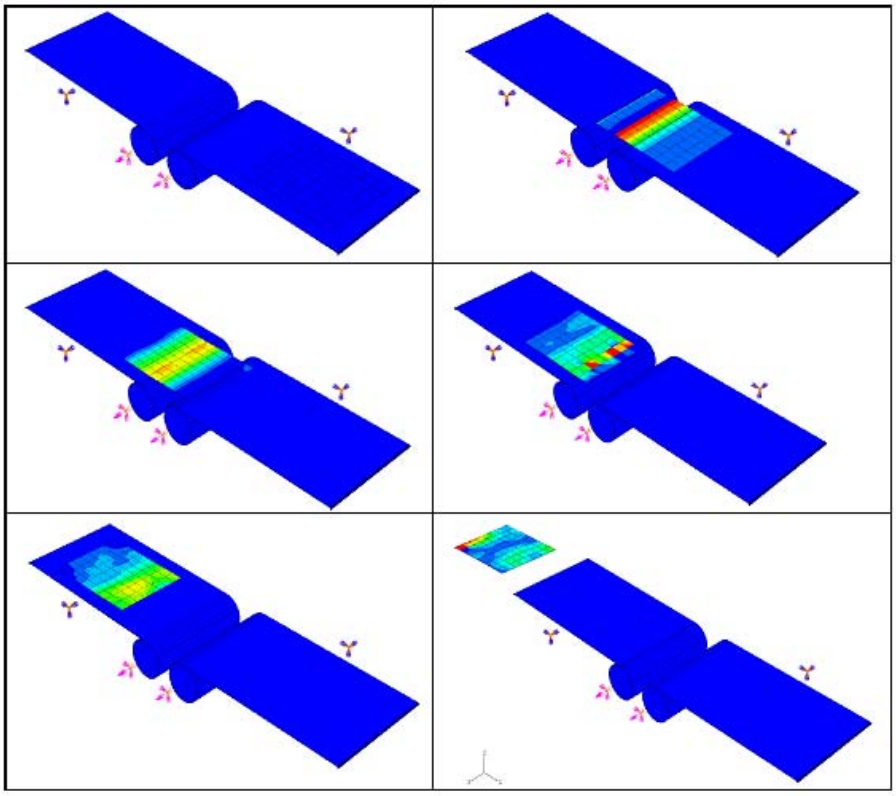
Paper sample with machine direction oriented 30° to the conveyor travel direction

The deflection values of the samples should increase as the orientation of the machine direction of the paper sample increase from 0° to 90° with respect to the conveyor travel direction. This is because of the lower values of the Young's modulus of the sample in cross-machine direction when compared to the machine direction.

RESULTS

Impact of Viscous Pressure:

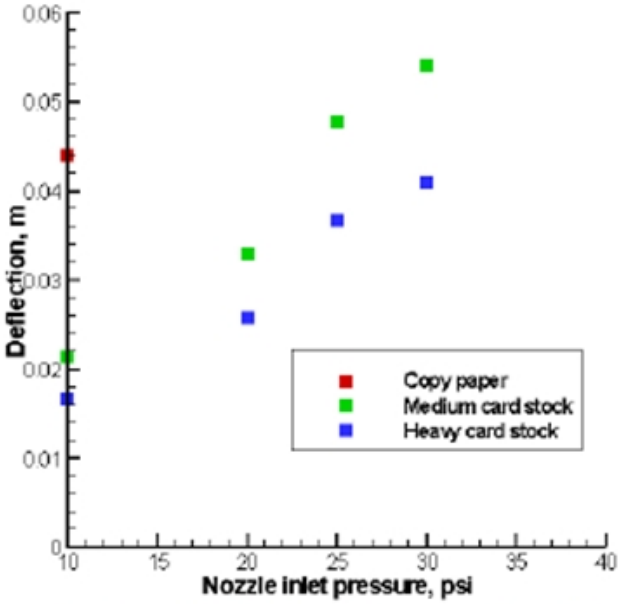
In order to identify the effects of the viscous pressure on the deflection of the paper sample, a finite element model was created with all the loading and boundary conditions as mentioned in the previous chapter except the viscous pressure and the external pneumatic load. The simulation results show that, the paper sample flies off the conveyor once it touches the conveyor on the other side. After some time, due to gravity the sample moves downwards. In the next simulation the viscous pressure was specified on the free surfaces of the paper sample. All the boundary and loading conditions are kept the same. The simulation result is shown in the following figure.



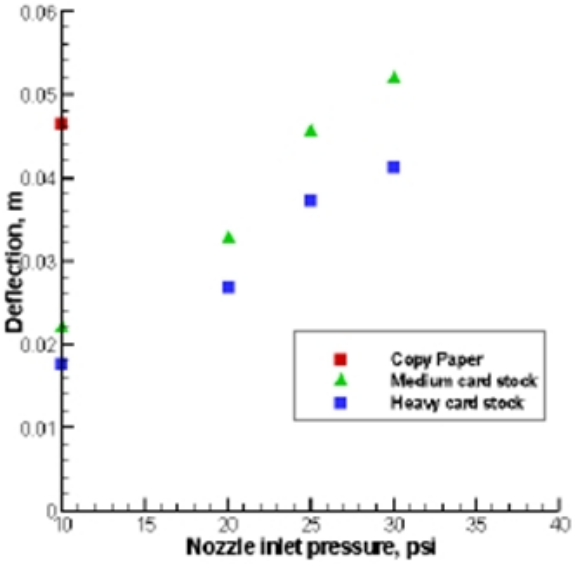
Response of the sample in the presence of viscous pressure

Simulation Results

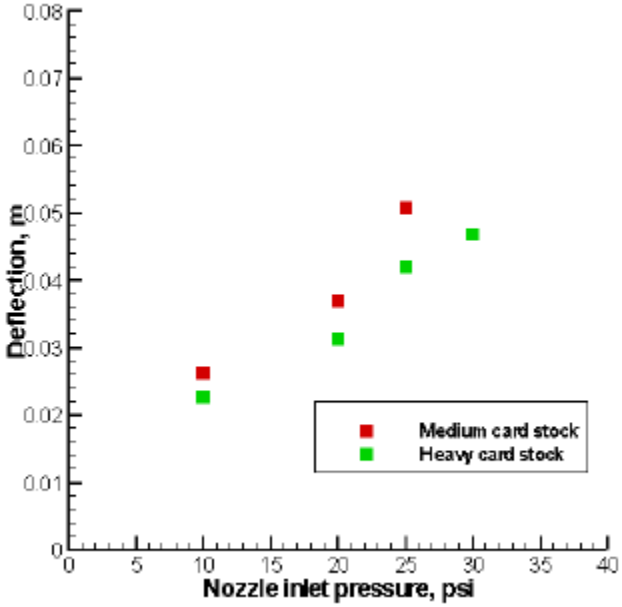
Two sets of simulations were run for two different conveyor speeds. For each speed, the sample deflections are found at each of the four nozzle inlet pressures and at different orientations of the paper on the conveyor. For first set of simulations, the conveyor speed is set to be equal to 300 ft/min. The analysis results are shown below



Samples oriented along the conveyor length



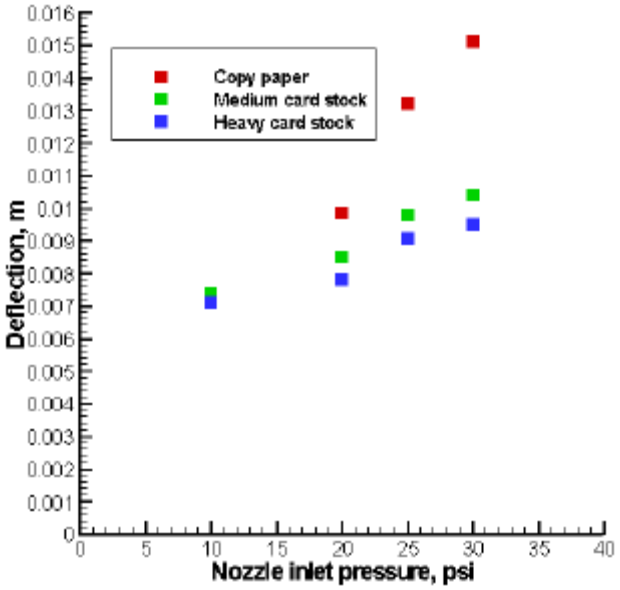
Samples oriented at 30° to the conveyor length



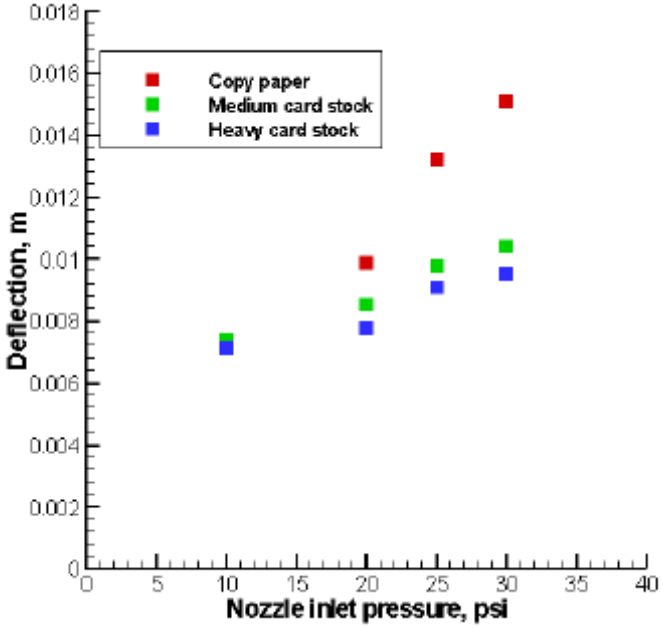
Paper sample oriented at 90° to the conveyor length

The copy paper was able to clear the gap only when it is traveling with its MD along the conveyor length, at 30° to the conveyor length and when the nozzle load is 10 psi. In all the other cases, the copy paper was unable to clear the gap.

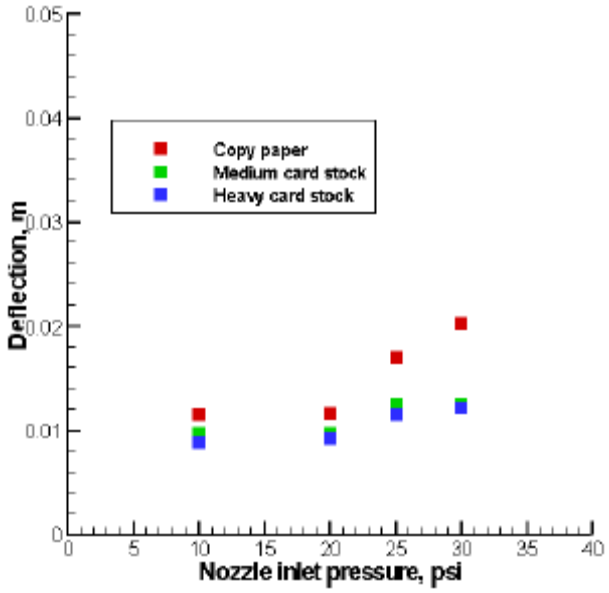
The speed of the conveyor is increased from 300 ft/min to the targeted speed of 1200 ft/min. The results of the simulation are shown below



Paper sample oriented along the conveyor length



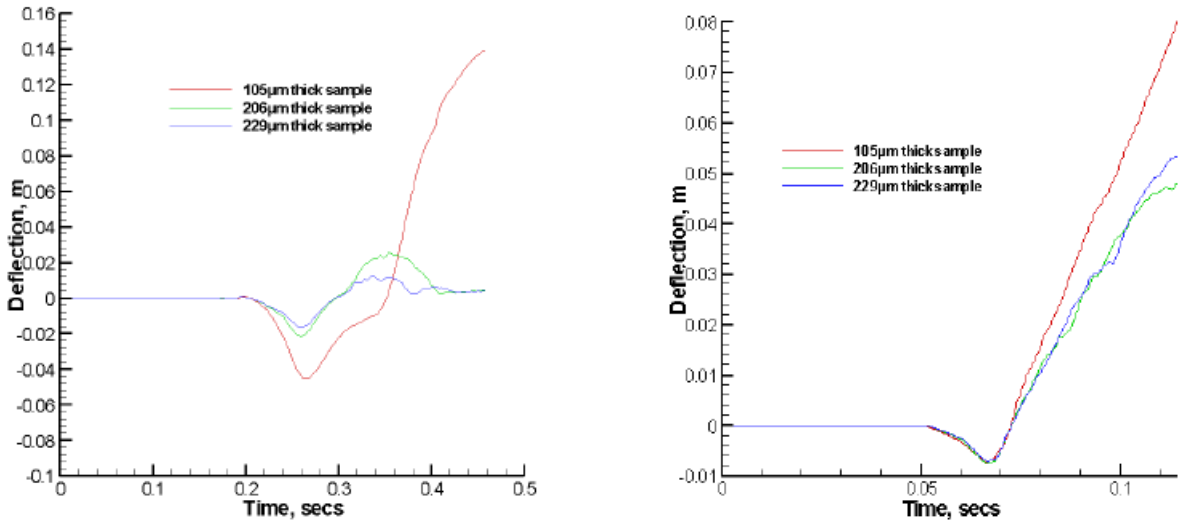
Paper sample oriented at 30° to the conveyor length



Paper sample oriented at 90° to the conveyor length

It is interesting to note that all the paper samples cleared the gap for all the four nozzle loads when the conveyor speed is increased to 1200 ft/min. At the same time, the deflection values of the paper samples decreased compared to the deflection values of the samples moving at 300 ft/min. This is expected because the samples moving at 1200 ft/min have high kinetic energy than the sample moving at 300 ft/min.

Another important aspect is the settling time the samples take to settle completely on the left conveyor surface after the load is applied. The samples moving at 300 ft/min are taking less time to settle on the conveyor surface than the sample moving at 1200 ft/min. This can be clearly seen from the figures shown below

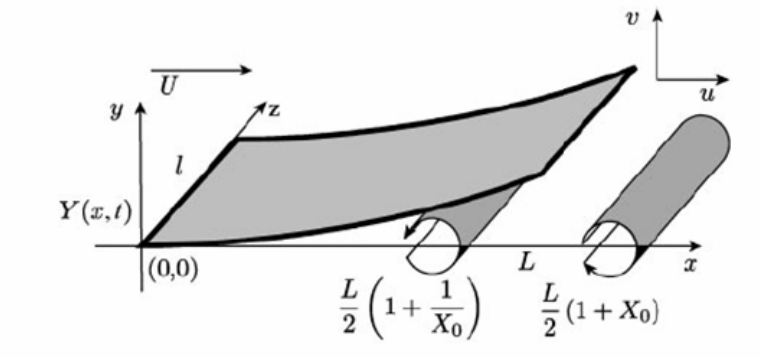


Response of samples moving at 300ft/m and 1200 ft/m

This behavior might be due to the lack of sufficient energy dissipation mechanism in the model. If the plasticity of the model is included more accurate results of the deflection can be obtained, this will also reduce the settling time of the samples at high speeds. This can be either done by using upgraded ABAQUS finite element analysis environment or by using FEA code from another vendor.

Flutter Based Stiffness Measurements.

One method to increase the robustness of stiffness sensing would be to measure the fluttering of the paper samples when they are excited. The work on *"Fluid Flow Induced Flutter of a flag" by Mederic Argentina and L. Mahadevan* has shown that the fluttering of a flag / a wind sail is directly related to the bending stiffness of the material. This is illustrated below.



When the velocity of the fluid U > $U_c = \left(Eh^3/\rho_f L^3\right)$

The frequency of flutter is given by $\omega = \left(\frac{Eh^2}{\rho_s L^4}\right)$, where

E	-	Young's Modulus
h	-	Thickness of the sample
L	-	Length of the sample
ρ	-	Density of fluid and the sample

The Bending Stiffness $D = \frac{Eh^3}{12(1-\upsilon^2)}$,where υ is the Poisson's ratio of the material

Hence we can arrive at a relationship between D and ω. However, most of the literature for this type of problem is based on the assumptions that

- The fluid flow over the sample is fully developed.

- The sample is fixed at one of the edges, (typically the leading edge into the flow).

Both these assumptions are not valid for our case. For the speeds at which the conveyor operates, it is not possible for a flow field to develop fully over a sample's length. And also, the sample's leading edge is not constrained by any means.

Hence, before we try to build a flutter based sensor, sufficient theory has to be developed which addresses the problems stated. A better part of the next quarter will be dedicated towards the understanding of the theory behind the flutter phenomenon as well as using FEA to simulate fluttering of the samples under the exact operating conditions as those expected on the conveyor.

PROGRESS TOWARDS SENSOR INTEGRATION

During the beginning stages of the integration process, we shall be using LabVIEW^{tm} for data acquisition and processing. This would help us in easier integration of different sensors and also help us better visualize the data from the sensors as compared to using the Motorola HC12 Microcontroller. Also more complex routines (Filtering, Higher order arithmetic etc...) can be implemented as computation time will no longer be an issue.

Lignin Sensor Modification

Earlier, the voltage data from the distance sensor and the Fluorescence Sensor were given as input to the microcontroller which then performed calculations based on the correction surface and returned a normalized lignin content value. It has now been modified to work with LabVIEW^{tm}. The Signals from the distance sensor and the fluorescence sensor are given to a Data Acquisition card, which is interfaced to LabVIEW^{tm}. All of the correction surface calculations (to compensate for the varying distance from sensor head) are integrated into the VI.

Advantages of Integration with LabVIEW^{tm}

The following changes are the advantages of using LabVIEW^{tm}.

- Automatic recalibration - The gain adjustment for the fluorescence sensor can be automated by placing known samples and let the software re-calibrate itself.

- Distance Correction calculations can now be upgraded to a fourth order surface which was not possible earlier with the microcontroller due to the restriction on computational time. This would result in higher accuracy in the lignin content estimation

- Enables software filtering so that any high frequency noise from both the distance sensor and the fluorescence sensor can be eliminated.

PLANS FOR NEXT QUARTER:

1. Develop the theory behind the fluttering phenomenon by taking into account the effects of an unconstrained leading edge and undeveloped flow fields.

2. Develop the Lignin sensor's VI by incorporating automatic recalibration, upgrading the distance sensor correction formulas to higher order surfaces.

3. Explore alternatives for high speed distance sensing for hard to distinguish grades.

References:

1. ABAQUS Version 6.4, analysis user's manual

2. Theory of Plates and Shells, S.Timoshenko, McGraw-Hill, Inc.

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4. Mann, R. W., Baum, G. A., and Habeger, C. C., 1980, ''Determination of All Nine Orthotropic Constants for Machine-made Paper,'' Tappi J., February, pp. 163–166.

5. Me´de´ric Argentina and L. Mahadevan, 2004,"Fluid-flow-induced flutter of a flag"

6. Nobuyuki Yamaguchi,Tooru Sekiguchi,Kazuhiko Yokota,2000,"Flutter Limits and Behavior of a Flexible Thin Sheet in High-Speed Flow",Journal of Fluids Engineering Mar 2000,Vol 22.

Patents: None

Publications/Presentations: None

Milestone Status Table:

ID Number	Task / Milestone Description	Planned Completion	Actual Completion	Comments
1	Design robust industrially worthy lignin sensor	12/03		In progress.
2	Develop color tracking system	12/03		In progress.
3	Develop a decision making algorithm	12/05		In progress.
4	Evaluate sensing techniques for food pkg, waxed OCC, coated OCC, high adhesive paper	12/05		In progress. The development of a stiffness sensor is towards this task.
5	Incorporate and test array of lignin/gloss/color sensors and decision-making algorithm.	12/06		

Budget:

521126	**2003**	**2004**	**2005**	**2006 Thru 06/2006**	**Cumulative thru 06/30/2006**
Total Direct Costs	24,630.81	103,101.39	146,983.27	268,470.84	268,470.84
Cost Sharing					
Venditti	329.15	874.77	966.65	5,908.26	8,078.83
Ramasubramanian	355.02	940.85	-	5,336.52	6,632.39
F&A on Cost Sharing	318.14	844.26	449.49	5,228.82	6,840.71
Total Cost Sharing	1,002.31	2,659.88	1,416.14	16,473.60	21,551.93
Third Party Cost Sharing					
Weyehauser(per Letter)	10,000.00	10,000.00	10,000.00		30,000.00
Advanced Sorting Technologies(per letter)	10,000.00	10,000.00	10,000.00		30,000
Total 3rd Party	20,000.00	20,000.00	20,000.00		60,000.00
Total Project Cost	45,633.12	125,761.27	168,399.41	284,944.44	350,022.77

Development of Renewable Microbial Polyesters for Cost Effective and Energy-Efficient Wood-Plastic Composites

Thompson: Idaho National Laboratory

Agr id:11428

QUARTERLY PROGRESS REPORT

Project Title: Development of Renewable Microbial Polyesters for Cost Effective and Energy-Efficient Wood-Plastic Composites

Covering Period: April 1, 2006 through June 30, 2006

Date of Report: July 28, 2006

Recipient:
Idaho National Laboratory
P.O. Box 1625
Idaho Falls, ID 83415

Project Number: CPS Number: 11428

Project Period: October 1, 2004 – September 30, 2008

Subcontractor:
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P.O. Box 643140
Pullman, WA 99164-3140

Other Partners:
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Glatfelter Corporation
Strandex Corporation
University of California-Davis

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Project Objectives:

Objective 1: Determine preferred PHA monomer compositions, PHA/cell debris ratios, and PHA/wood ratios for the production of superior wood-PHA composites

Objective 2: Define feedstock compositional ranges for WTE and PPE for production of PHAs meeting PHA monomer composition ranges specified in Objective 1

Objective 3: Determine the efficacy of supplementing WTE and PPE to improve PHA production from these effluents

Objective 4: Test the material properties of wood-PHA composites produced from waste-derived PHA made and used without extraction or purification

Objective 5: Produce and test wood-PHA composites made from PPE-derived PHA at the pilot-scale.

Background:

This is a collaborative project among the Idaho National Laboratory, Washington State University, the University of California-Davis, Glatfelter Corporation, Strandex Corporation, and ECO:LOGIC Engineering, Inc. The purpose of the project is to develop and produce wood-plastic composites using bacterial polyhydroxyalkanoates (PHA) in place of petroleum-derived plastic. This will be made economical and environmentally friendly by reducing or eliminating costly steps in PHA production. Specifically, this will be accomplished by utilizing PHA produced inherently in wastewater treatment processes, and by utilizing the PHA directly in the composite without removal of the cell debris. The forest products industry will benefit most from this research, both from the perspectives of environment, recycling, and new composite products. The municipal waste treatment industry, as the basis for production of PHAs from effluents, provides the starting point for application to forest products industry effluents and also benefits.

This project is comprised of five tasks. The first four tasks address PHA production, extrusion, and composite properties. Laboratory testing to determine preferred feedstock compositions, together with ease of processing and material properties of composites produced therefrom, will be completed by WSU for pure commercial PHAs (Task 1) and for unpurified effluent-derived PHAs (Task 4). This information will be used to define appropriate effluent feedstocks (Task 2) and optimize supplements (Task 3) to support the preferred composite formulations. Task 5 will include a ca. 1000 lb/h pilot-scale extrusion of wood-WTE-derived PHA composites using the appropriate supplements in the production of the WTE utilized. The project is being completed in three phases. Phase 1 includes determination of PHA compositions and amounts for superior wood-thermoplastic composites. Phase 2 includes determination of feedstock supplements for production of effluent-derived PHAs of the desired monomer compositions. Phase 3 includes pilot-scale testing to identify and mitigate processing issues at the commercial scale.

Status:

Tasks scheduled to be active during this quarter included

- Task 1, "Purified PHA ± Cell Debris Composite Processing & Material Properties,"
- Task 2, "Effect of Feedstock on PHA Type/Amount,"
- Task 3, "Supplementation of Waste Effluents for Production of PHA," and
- Task 4, "Waste Effluents PHA Composite Processing & Material Properties."

Task 1 and Task 4 work scopes are being performed at Washington State University (WSU). DOE funded Task 2 and Task 3 work scopes are being performed at INL, UC-Davis, ECO:LOGIC, Inc., and Glatfelter. Highlights of work performed during this quarter include:

- The attainment of greatly improved mechanical properties for the wood-PHA composites with the use of appropriate lubricants and compatibilizers (Task 1), and
- Successful production of PHA by pulp mill-derived microbes using pulp mill wastewater as the carbon source (Tasks 2 & 3).

Task 1 Status:

Experiments were performed to determine the effects of processing additives on mechanical properties, including lubricants and compatibilizers. In addition, a study was done to determine the effect of extrusion screw speed on strength and quality. The shear viscosity of a polymer composite melt increases quickly with fiber (filler) content. As in other WPCs, lubricants are necessary in PHB/wood fiber composites having high fiber contents in order to allow high speed extrusion and quality surface characteristics. Several commercial lubricant products were compared (Figure 1), including N,N-ethylene bissteramide (EBS), zinc Sterate (66%)/EBS (33%), partially saponified montanic ester wax (OP100), oxidized Polyethylene, polyethylene wax (Struktol 306), and amide lubricant (Glycolube WP2200). Results indicated that Glycolube yielded the highest flexural properties of the composites, which were substantially higher that the rest of the lubricants (Figure 1).

Compatibilization experiments focused on the use of methylene diphenyl diisocyanate (MDI). In spite of polar ester linkages and functional end groups in the molecule, PHB is highly hydrophobic. Wood fiber, on the other hand, is composed primarily of cellulose and hemicellulose and its surfaces are highly hydrophilic. Figure 2 demonstrates that the overall flexural properties increase with addition of methylene diphenyl diisocyanate (MDI) up to 5% in the composites. Particularly, flexural strength increased by more than 100% with only 4% MDI addition, and modulus and strain at break also had large increases. This result has been shown previously for composites composed of biopolyesters and natural fiber. It has been shown in the literature that the mechanical properties of PLA/bamboo fiber composites were improved significantly by coupling agents. Other evidence from the literature showed that interfacial adhesion between polycaprolactone and wood was improved by adding compatibilizers, and adhesion between PHAs and flax was clearly improved by fiber surface modification using silane coupling agents.

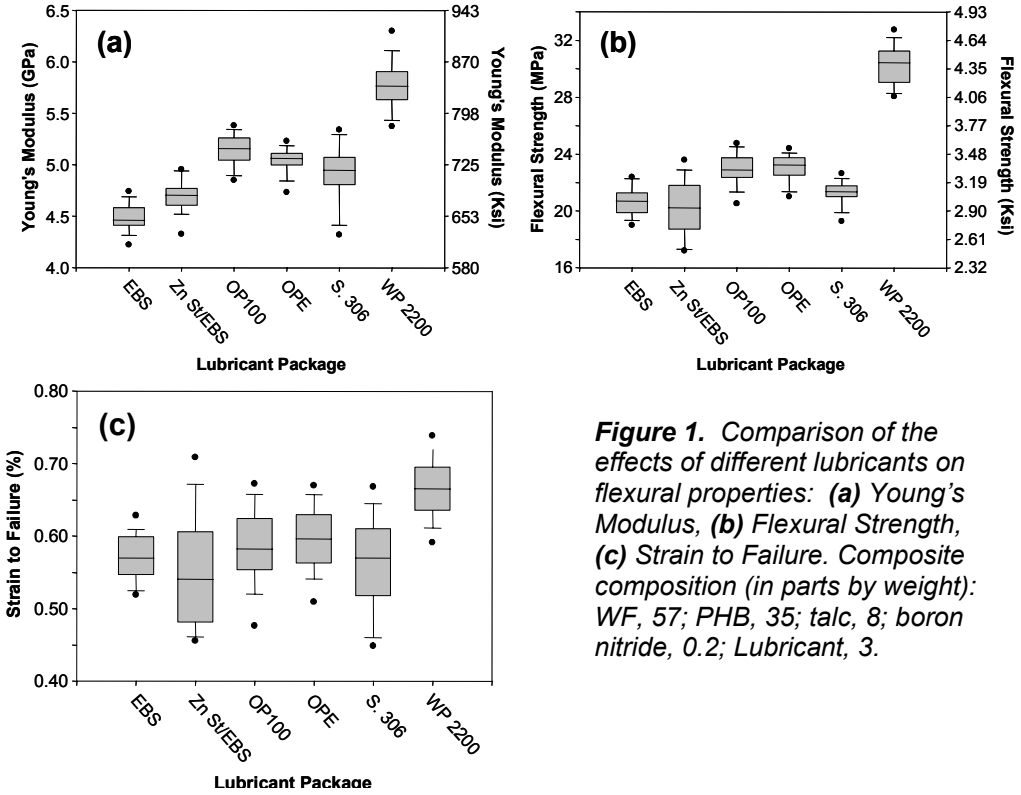

Figure 1. *Comparison of the effects of different lubricants on flexural properties:* ***(a)*** *Young's Modulus,* ***(b)*** *Flexural Strength,* ***(c)*** *Strain to Failure. Composite composition (in parts by weight): WF, 57; PHB, 35; talc, 8; boron nitride, 0.2; Lubricant, 3.*

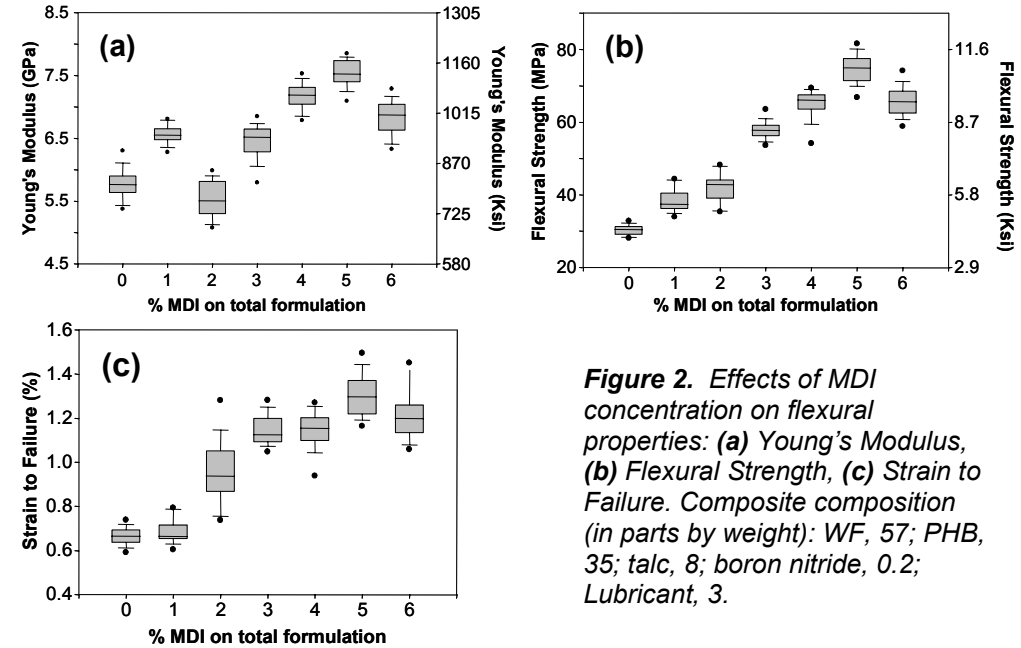

Figure 2. *Effects of MDI concentration on flexural properties:* ***(a)*** *Young's Modulus,* ***(b)*** *Flexural Strength,* ***(c)*** *Strain to Failure. Composite composition (in parts by weight): WF, 57; PHB, 35; talc, 8; boron nitride, 0.2; Lubricant, 3.*

Finally, the effect of extrusion screw speed was investigated, as it is desirable to utilize high speed processes for industrial scales. Particularly for PHB, because it is susceptible to thermal degradation during processing, reducing the residence time while maintaining extrudate quality is critical. The reduction of residence time can be achieved by increasing the screw speed. Figure 3 shows the results of a comparison of flexural properties of extrudate at screw speeds of 10 and 15 rpm. Both MOE and strain at break were higher at 15 rpm screw speed (shorter residence time) than at 10 rpm, but MOE did not show a clear difference at these two speeds. However, extrudate obtained at a screw speed of 5 rpm produced extrudates with considerably inferior mechanical properties (data not shown).

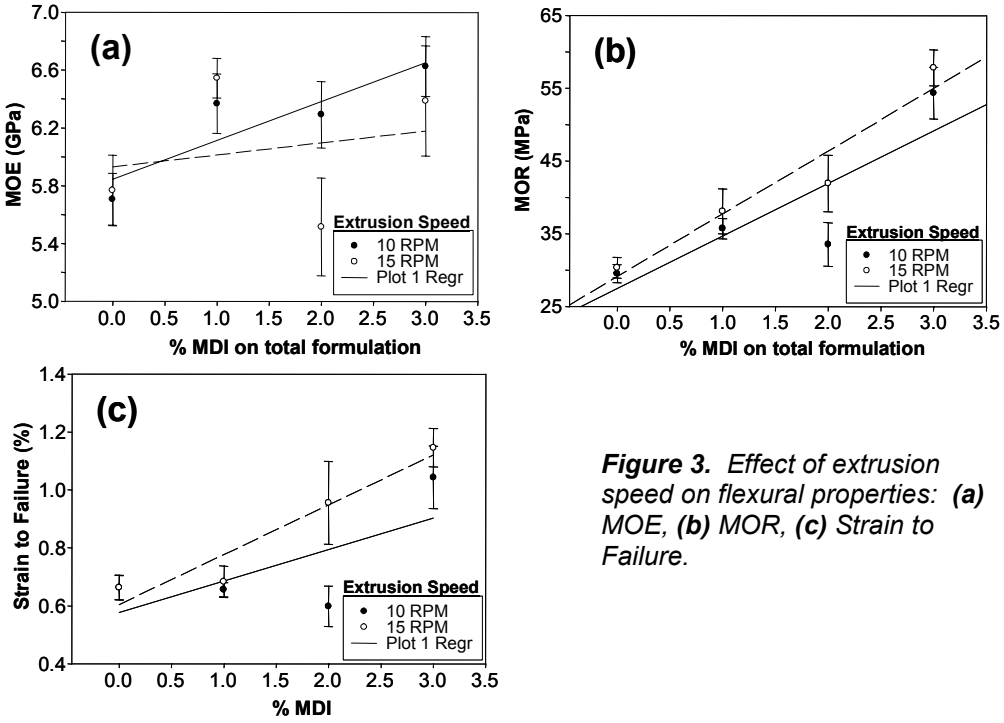

Figure 3. *Effect of extrusion speed on flexural properties:* ***(a)*** *MOE,* ***(b)*** *MOR,* ***(c)*** *Strain to Failure.*

Tasks 2 & 3 Status:

Bench scale reactor operations continued at UC-Davis throughout the quarter. All four Food:Microorganism (F:M) ratios (.5:1, 1:1, 2:1, and 3:1) were examined for each of the main reactors in operation. The reactors are all on 24-hour 6 hour anoxic, 18 hour aerobic cycle with the following specifications:

A. 100% fermentate fed , fed at end of anaerobic cycle, MLVSS 1900-2100 mg/L
B. 100% fermentate fed, fed at beginning of anoxic cycle, MLVSS 1800-2000 mg/L
C. 50% fermentate fed, fed at beginning of anoxic cycle, MLVSS 1000-1200 mg/L
D. 25% fermentate fed, fed at beginning of anoxic cycle, MLVSS 600-800 mg/L
E. 10% fermentate fed, fed at beginning of anoxic cycle, MLVSS 400-600 mg/L

Figure 4 is a comparison of the PHA quantities present in the main reactors over the 24 hour cycle represented as a percentage of MLVSS on a dry weight basis. The results indicate that the greater the fermentate concentration within a reactor, the greater the amount of PHA present in the reactor. The quantity of PHA in reactor E was below the detection limit.

Figure 5 shows a comparison of the peak PHA percentage in side stream reactors operated from each of the main reactors for a 2:1 F:M ratio. PHA percentage appears to go down as the main reactor fermentate concentration, synonymous with MLVSS, goes up. However, if these results are analyzed as a yield in g/L instead of a percentage, the opposite becomes true (i.e. you will get a higher quantity of PHA out of the reactors with higher MLVSS). If a minimum of 20% PHA is the only requirement, and is just as sufficient as 30% or higher, then a reactor with higher MLVSS can be used. If the main goal is to the highest percent PHA possible, then the reactors with the lowest MLVSS will have to be used. A side note: Hour 8 of reactor E was an incomplete sample, so it is possible the peak should be there. The pH and COD data collected at that point also indicate hour 8 as the likely location for the peak.

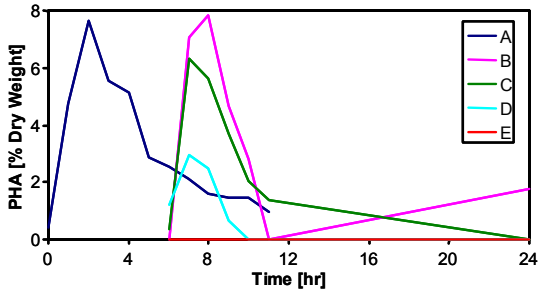

Figure 4. *PHA percentage present in the main bench scale reactors over a 24 hour time period on a percent dry weight basis.*

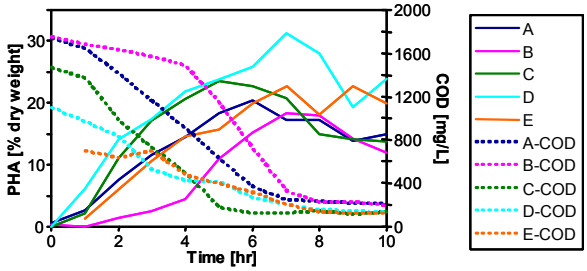

Figure 5. *Comparison of 2:1 F:M ratio side stream reactors operated from each of the five main reactors.*

Figure 6 shows a comparison of PHA percentages obtained for the four different F:M side stream reactors from reactor B. PHA percentage clearly increases with F:M ratio. Initial test lasting 12+ hours showed no increase or peak in PHA for the 3:1 F:M ratios. Additional testing performed at the end showed this peak had occurred sometime between 18 and 30 hrs. This was concluded by testing done at 30hrs which showed an increase in pH and an eventual decrease in COD. No data for the PHA percentages were collected in that interval. The online monitoring of the pH and DO will eventually help to more closely identify where the peak is.

Figure 7 shows the relationship between pH, dissolved oxygen (DO), and PHA. These results are from a side stream from reactor C with a 2:1 F:M ratio. The relationship between the parameters is typical for side stream reactor operation. In the figure, the pH peaks initially and then goes back down and will eventually go back up again. This initial peak corresponds to the peak in PHA. This happens consistently in all of the side stream testing. This is very important because each batch of wastewater is different. The MLVSS can vary within the reactors, and the COD will vary within the fermentate as a function of the water content in the primary solids. All of these things can affect when the PHA is going to peak. If we have a way to monitor this

real time then we know when to extract the sample. Additionally, DO demand increases noticeably when the bacteria begin to utilize COD. The spike in DO, along with the increase in pH, is an excellent indicator of when the microbial consortia will start producing PHA.

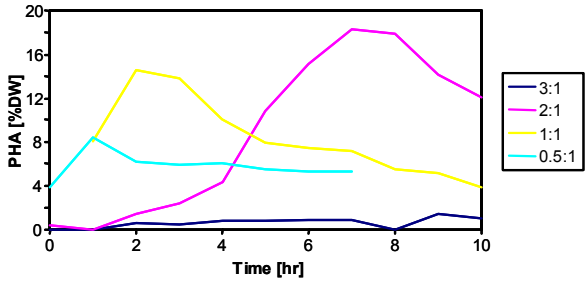

Figure 6. *Comparison of peak PHA percentages four the four F:M side stream reactors operated using reactor B as the microbial source.*

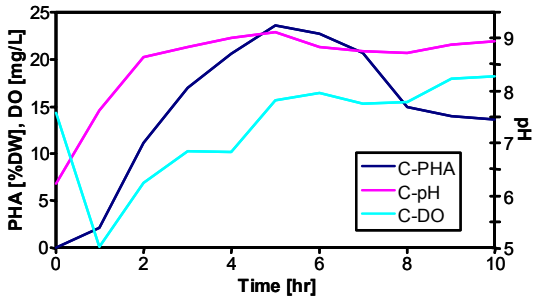

Figure 7. *Correlation between PHA, DO, and pH in a typical side stream reactor.*

Pulp mill based work on Tasks 2 and 3 has progressed at INL on identifying suitable carbon sources within a pulp mill that can be used to feed pulp mill-derived activated sludge for PHA production. Based on our prior consultation with the Glatfelter mill in Chillicothe about accessible mill-derived waste streams, our focus is on several pulp mill derived effluents (PPE), including foul condensate streams (FC) and the primary wastewater effluent (PWE). Overall, the specific feedstocks govern the PHA content and composition, whereas improper nutrient balance and toxicity in these effluents can hinder the mixed microbial consortia from growth and/or PHA accumulation in the biomass. To determine the efficacy of supplementation of these waste streams for PHA production, the experiment was broken into two stages. Stage 1 included characterization of the FCs and the PWE. Phase 2 included supplementation of selected FCs and PWE for PHA production.

To define the appropriate mill-derived waste streams, the water quality parameters for several FCs and PWE were analyzed. As expected, FCs had extremely high COD values (e.g. COD of FC#3 was 34,650 mg/L), whereas PWE had relatively low COD values (e.g. 570 mg/L). Both the FCs and the PWE had relative high COD/N ratios, which indicates that nutrient addition would be required. GC/MS scanning analysis of the FCs indicated methanol as the primary source of carbon for the microbes. In contrast, GC/MS scanning analysis of the PWE indicated that of the compounds contributing to the COD, potential carbon sources for the microbes included only 2-butynoic acid, acetic acid, and similar compounds. Other compounds that would likely not serve as carbon sources included benzene, acetone, methylene chloride, and chloroform, which are potentially toxic to the microbes. Hence, depending on the carbon source, not all of the COD present will be assimilable by the microbial consortia used.

To illustrate the feasibility for using FCs to feed pulp mill-derived activated sludge for PHA production, several streams were collected from various points within the foul condensate

collection system at the Glatfelter mill in Chillicothe, and were individually tested for microbial toxicity. Seven sampling locations were selected:

1) Evaporator foul condensate tank
2) High volume/low concentration (HVLC) condensate seal pot
3) Kraft mill foul condensate
4) Blow heat accumulator overflow (BHAO)
5) Evaporator combined condensate
6) Stripped condensate
7) Combined clean condensate

Methanol concentrations at each of these sampling points ranged from 10 g/L in the Kraft mill foul condensates, to 0.3 g/L in the HVLC condensate seal pot and the Evaporator combined condensate. A microbial enrichment was formed by inoculating a non-selective medium (trypticase soy broth) with secondary solids from the wastewater treatment facility at the Glatfelter mill. This enrichment, referred to as the Glatfelter consortia, was used to evaluate the potential growth inhibition of each of the condensates on a mixed microbial culture derived from activated sludge from the mill's wastewater treatment facility. A standard antimicrobial sensitivity test—the Kirby-Bauer method—was modified to test the condensates using a mixed microbial culture. Somewhat surprisingly, none of the condensates inhibited growth of the Glatfelter consortia at either concentration.

A subsequent test was performed using Glatfelter activated sludge consortia grown in a dilute mineral medium using each of the condensates as a potential carbon and energy source. Condensates were supplied at 10 and 25% and cultures were grown for 30 days. Growth was periodically monitored by optical density measurements. After 30 days growth was observed in all condensates, however maximum growth was observed in samples containing Kraft mill foul condensate, BHAO condensate, and Evaporator foul condensate at 25%. It is notable that these three condensates contain some of the highest methanol concentrations within the condensate collection system: 10, 4.3, and 2.9 g/L, respectively. These three condensates were selected for further investigation described below.

To determine the efficacy of supplementation of selected FC and PWE for PHA production, waste streams utilized included:

- Kraft mill foul condensate (FC) adjusted to pH 7-8
- Primary wastewater effluent (PWE)
- Primary wastewater effluent with reagent-grade methanol added to 8 g/L and 2 g/L

Four sequencing batch reactors (SBRs) fed with FC were run using a 2^2 factorial design varying nutrient additions and oxic-anoxic cycling, with Sludge Retention (SRT) and Hydraulic retention (HRT) times equal at 4 days (see Table 1). During operation, pH values were adjusted to 7 daily. All of the systems failed with regard to COD degradation, and were characterized by MLVSS wash out.

Table 1. *Experimental design matrix for PHA production using foul condensates.*

	Reactor R	Reactor O	Reactor C	Reactor K
Nutrient addition	+	+	-	-
SRT & HRT (days)	4	4	4	4
Anoxic-oxic cycling	-	+	-	+

For the PWE, 2 SBRs were run at HRTs of 1 and 4 days, and an identical 4 day SRT condition. Once the performance of these 2 main reactors stabilized (12 days), 2 additional batch tests were operated under conditions similar to the parent SBR fed with PWE, except at higher organic loading (see Table 2). No PHA was detected in the batch test operating in parallel with the main SBR at 1day-HRT or in the main SBR at 4 day HRT.

However, approximately 11% PHA content on a wet cell basis was obtained from the main SBR at 1 day HRT (Table 2). This value is on a wet cell basis; on a dry cell basis the value will be higher due to the removal of the excess water weight (we have not yet determined the dry cell percentage PHA for this experiment). Note that this result meets and exceeds Milestone 2.4, which requires ≥ 1% of the dry cell mass in PHA to be produced from pulp mill effluents using pulp mill bacteria. The percentage PHA in the cells can be increased further with appropriate addition of extra carbon, which we are exploring and will continue to explore through the use of supplemental carbon from various side streams produced at the mill, e.g. the foul condensates. As stated above, it is surmised that the methanol in the foul condensates could be a main carbon source for the mixed microbial consortia. Consequently, 1 SBR inoculated with the mill's activated sludge and fed with PWE spiked with 8 g/L methanol and operated with an HRT/SRT

Table 2. *PHA production from primary effluent under 4day-HRT and 1day-HRT condition*

Time (hr)	reactor A PHA (%)		Time (hr)	reactor C PHA (%)	
	main reactor	batch test		main reactor	batch reactor
initial F/M	3.5 d^{-1}	11.5 d^{-1}	initial F/M	1.3 d^{-1}	4.9d^{-1}
0.0	nm	0.0	0	nm	nm
0.5	8.9	0.0	1	0.0	nm
1.0	11.2	0.0	2	0.0	nm
1.5	10.0	0.0	3	0.0	nm
2.0	11.7	0.0	4	0.0	nm
2.5	10.7	nm	5	0.0	nm
3.0	nm	nm	6	nm	nm
3.5	11.4	nm	8	nm	nm
4.0	10.1	nm	9	nm	nm
4.5	11.5	0.0	10	nm	nm
5.0	12.1	nm	11	nm	nm
			12	nm	nm
			13	nm	nm
			15	nm	nm
			24	nm	nm

nm = not measured because samples were damaged during shipping to UC-D

of 4 days, and under fully aerobic conditions to enrich for microorganisms capable of producing PHA while concurrently degrading methanol. The system initially failed with regard to COD degradation and MLVSS wash out and was subsequently shut down. The failure was assumed to be due to excessive levels of methanol, however other factors such as time needed to increase the population of methylotrophs (methanol oxidizers) or their mineral nutrient requirements (such as Zn, Mg, and Cu) were not considered. Future experiments will address these concerns.

A lower methanol level experiment was performed where 1 SBR was inoculated with the activated sludge and fed with PWE spiked at 2g/L methanol and operated with an HRT/SRT of 4 days under fully aerobic conditions. The system was operated for 10 days but was prematurely shut down due to technical difficulties. Note that for methanol enrichment/selection, the SBR should be operated for a minimum of a 1-month period to allow selection for methylotrophs.

Task 4 Status:

Task 4 work at WSU is currently on hold due to insufficient funds availability to support this activity.

Plans for Next Quarter:

For Task 1, Rheological and morphological characterizations of the lubricated and compatibilized composites will be conducted.

For Task 2 work focused on completing the tests using municipal activated sludge consortia as inoculum source, the set of reactors operating with municipal wastewater as the feedstock have been shut down for now. It would be beneficial to further investigate how high the percentage of PHA could go with higher F:M ratios now that there is a way to determine where that peak should fall. In the past, arbitrarily choosing a testing interval proved difficult as the side stream reactor eventually lost enough volume that stable operation was difficult. Additionally, the decrease in MLVSS as the concentration of fermentate increases in a side stream reactor must be accounted for. Experiments need to show what percentage of PHA provides a stable NFRTC product, and whether the quantity or quality of the dried biomass is more important.

For Tasks 2 and 3 work focused on PHA production by pulp mill activated sludge consortia, we successfully produced PHA from pulp mill primary wastewater effluent (PWE), using pulp mill-derived microbes. Future work will focus on identifying operational parameters influencing the proportion of PHA and characterizing specific functional relationships of key operational parameters influencing the proportion and quantity of PHA produced. SBRs will be operated with mill-derived activated sludge under different levels of external acetate addition, HRT, and SRT, to further characterize the specific relationship between the listed parameter values and the proportion of PHA in cellular biomass. For the supplementation with foul condensates, we will start with selectively enriched, mill-derived activated sludge consortia capable of growth on methanol and FC under controlled nutrient conditions. A Master's student from UC-Davis,

Gregory Mockos, began work at the INL in mid-June to accomplish this task. Specific streams to be utilized for the enrichments include: Kraft mill foul condensate, BHAO, and Evaporator foul condensates, Methanol, and Primary clarifier effluent and supplemented with methanol. Each carbon source will be supplemented with a nutrient medium to eliminate the possibility of selection based on nutrient limitations. Based on previous tests, it is expected that the enrichments will require on the order of 60 days to select for the appropriate microbial communities. The enrichments will be used to construct a consortium based on Glatfelter's activated sludge that can produce PHAs from primary clarifier effluent that has been supplemented with one or several of the foul condensates. These consortia will subsequently be tested for their abilities to produce PHA in FC-fed SBRs run under various operational conditions of HRT and SRT to optimize PHA production.

Task 4 work at WSU is currently on hold due to insufficient funds availability.

Patents:

No patents resulting from the project were awarded or applied for during this quarter.

Publications/Presentations:

No presentations were given or publications issued during this quarter resulting from the project.

Milestone Status Table (as of June 2006 accounting month end):

ID Number	Task / Milestone Description	Planned Completion	Actual Completion	Comments
1.0	**Task 1 – Purified PHA ± Cell Debris Composite Processing & Material Properties**			
1.1	Physical & rheological properties of PHAs and WFRTCs defined	3/31/06		Delayed due to funding shortfalls
1.2	Composite processing/mechanical properties of pure PHA WFRTCs completed	7/31/06	6/30/06	
1.3	Wood/purified PHA composites with integrated cell debris produced having MOR ≥ 1500 psi and MOE ≥ 0.20 Mpsi	8/4/06	12/31/05	Met and exceeded
2.0	**Task 2 – Effect of Feedstock on PHA Type/Amount**			
2.1	WTE survey of several waste treatment facilities completed	7/1/06	6/30/06	Further tests useful but not critical
2.2	Enriched paper mill inoculum source and/or ATCC Sphaerotilus culture ready for testing	11/30/06		
2.4	PHA produced from a PPE source by indigenous or inoculated laboratory cultures at ≥ 1 wt% of the dry cell mass	4/1/07	6/5/06	Met and exceeded
2.3	Unsupplemented PHA from PPE completed	8/1/07		
3.0	**Task 3 – Supplementation of Waste Effluents for Production of PHA**			
3.1	PPE supplements & production criteria for pilot test defined	12/15/07		
3.2	In situ PPE process requirements for pilot test defined	12/15/07		
4.0	**Task 4 – Waste Effluents PHA Composite Processing & Material Properties**			
4.1	Material & properties defined for waste-PHA composites	815/07		
4.2	Wood/purified PHA composites with integrated cell debris produced having MOR ≥ 2000 psi and MOE ≥ 0.25 Mpsi	8/15/07		
4.3	Basic processing conditions defined for pilot test	10/15/07		
4.4	Formulations identified for pilot extrusions	4/1/08		
5.0	**Task 5 – Pilot-scale Extrusion Testing of Waste Effluents PHA Composites**			
5.1	Pilot test plan completed	12/15/07		
5.2	Supplemented or unsupplemented PPE biosolids produced for pilot extrusions	2/15/08		
5.3	Pilot extrusions completed	7/15/08		
5.4	Project completion and transition planned to technology demonstration phase	7/31/08		
5.5	Final Report delivered to DOE	9/30/08		

Budget Data (as of June 2006 month end):

Phase / Budget Period			Approved Spending Plan			Actual Spent to Date		
			DOE Amount	Cost Share	Total	DOE Amount	Cost Share	Total
	From	To						
Yr 1	10/1/04	9/30/05	$262,000	$305,411	$567,411	$260,204	$334,364	$594,568
Yr 2	10/1/05	9/30/06	$225,000[a]	$459,726	$684,726	$194,787	$261,486	$456,273
Yr 3	10/1/06	9/30/07	$550,514	$482,006	$1,032,520			
Yr 4	10/1/07	9/30/08	$402,486	$137,400	$539,886			
Yr 5								
		Totals	$1,440,000	$1,384,543	$2,824,543	$454,991	$595,850	$1,050,841

a An amount of $1,797 was carried over from FY2005 to FY2006 and is not included in this figure.

Rapid, Low Temperature Electron, X-Ray, and Gamma Beam Curable Resins

Griffith: Oak Ridge National Laboratory

Agr id:10276

QUARTERLY PROGRESS REPORT

Project Title: Rapid, Low-Temperature Electron, X-ray, and Gamma Beam-Curable Resins

Covering Period: April 1, 2006 through June 30, 2006.

Date of Report: July 31, 2006

Recipient: Oak Ridge National Laboratory
Managed by UT Battelle, L.L.C for the Department of Energy
Post Office Box 2008
Oak Ridge TN 37831-6150

Award Number: DE-AC05-00OR22725

Subcontractors: P. M. Winistorfer, Department of Wood Science and Forest Products, Virginia Tech

G. F. Dorsey, W. W. Moschler, Jr., T. G. Rials, and D. P. Harper, Tennessee Forest Products Center, University of Tennessee

Other Partners: J. B. Eppner, Dow Chemical Company, 2301 N. Brazosport Blvd., Freeport, TX 77451.

L. A. Parks and Cheng Song, SteriGenics International, Inc., 7695 Formula Place, San Diego, CA 92121-2418.

A. G. Landers, J. M. Huber Corporation, Engineered Woods, P. O. Box 670, Commerce, GA 30529.

J. Fyie, TrusJoist, a Weyerhaeuser Business,2910 East Amity Road, P. O. Box 8449, Boise, ID 83707-2449.

Contact(s): William L. Griffith, (865) 574-4970, griffithwl@ornl.gov

Project Team: Project Team: Drew Ronneburg, DOE program manager; Wood and Wood Composites Task Group (mentors).

Background: Approximately 50% of all wood used today is some type of glued-wood assembly. The manufacture of most glued-wood assemblies requires process heat. Process heat is required to dry the parent wood material, assist in consolidation of the product (flat-pressed panel products) and polymerize and cure the resin system. Glued-wood products range from structural laminated beams and flat-pressed panels to furniture assemblies and non-structural wooden assemblies. Many of these products are referred to as wood composites. The moisture content of the wood materials must be reduced to low levels and then be carefully controlled to ensure efficient resin polymerization and eventual resin curing and to avoid generation of excessive steam vapor pressure internal to the product. Drying the wood furnish materials and controlling the substrate moisture content is a major consumer of energy in the manufacturing plant. "Hot-pressed"

wood panels such as oriented strand board, medium density fiberboard (mdf) and particleboard can be mismanufactured by lack of moisture control.

Development of rapid, low-temperature electron beam-curable resin systems offers an **energy savings potential to the wood composites industry of 65 Trillion Btu/yr at full market penetration.** The lower curing temperatures (from 450 °F to 250 °F) possible with beam-curing systems also offer the potential of reducing unit capital costs and doubling throughput. The **lower curing temperatures can also decrease process emissions by reducing organic volatiles**.

Status: Discussions have been held with each of the industrial partners and options for commercialization have been discussed. These discussions will continue. Tentative plans are to hold the technology transfer meeting in the October- November time frame to attract the largest attendance.

Previous results demonstrated a significant reduction in mechanical strength on the wood at and above a dose level of 80 kGy with no impact on bending modulus. Currently, the investigation is focused on the impact of the e-beam on the viscoelastic properties of the wood. This investigation can point to specific mechanisms that are causing degradation of strength properties.

Dynamic mechanical analysis has been conducted with irradiated wood specimens being submerged in a bath of ethylene glycol. The purpose of the bath is to reduce the complete glass transition temperature of wood lignin to below a temperature where degradation of wood occurs. From the results, a decrease in the a-transition of wood lignin is observed (Table 1). However, a noticeable change in the magnitude of the storage (Figure 1) and loss (Figure 2) modulus curves was not observed. There is likely a change in the molecular weight and scission of side groups allow for increased mobility of the lignin molecules. The chemical and physical mechanisms for the behavior will be investigated further with ongoing infrared spectroscopy and additional analysis of the irradiated wood specimens.

Previously, wood-polyethylene-methacrylate composites were reported to show improved stiffness, strength, toughness, and moisture resistance when exposed to 80 kGy of radiation. Electron microscopy was performed to image the location and distribution of the methacrylates in the composite. Using primary electron (backscattering) imaging, submicron sized zinc particle were observed to be well distributed and in close proximity to the wood filler in the polyethylene matrix (Figure 3). This helps bolster the hypothesis that the zinc will be attracted to the wood surface and the methacrylate may produce bonds to the wood fibers.

Infrared spectroscopy is being conducted on wood-polyethylene composite to determine if there is bonding between the wood substrate and the zinc acrylate. In addition, further trials are being planned and materials have been acquired to investigate the impact of radiation dose, metal-acryl ate loading, and the use of high-density polyethylene on the physical and mechanical properties of the composite.

Table 1: Maximum loss modulus, E″ for each of the measured frequencies at each dose level. (The activation energy, E_a is obtained from an Arrhenius plot of the shift factors.)

Dose (kGy)	20	10	5	2	1	0.5	0.2	0.1	E_a (kJ/mol)
0	76.19	70.84	66.82	62.76	60.21	57.06	54.35	52.09	260
40	71.05	66.61	63.17	59.40	56.89	53.67	50.66	48.43	265
80	68.30	64.03	60.67	56.86	54.40	51.66	49.38	47.97	260
120	61.67	56.43	51.72	45.70	41.61	37.16	35.78	35.24	234
180	59.45	54.85	51.09	46.81	43.94	39.99	37.16	35.87	253

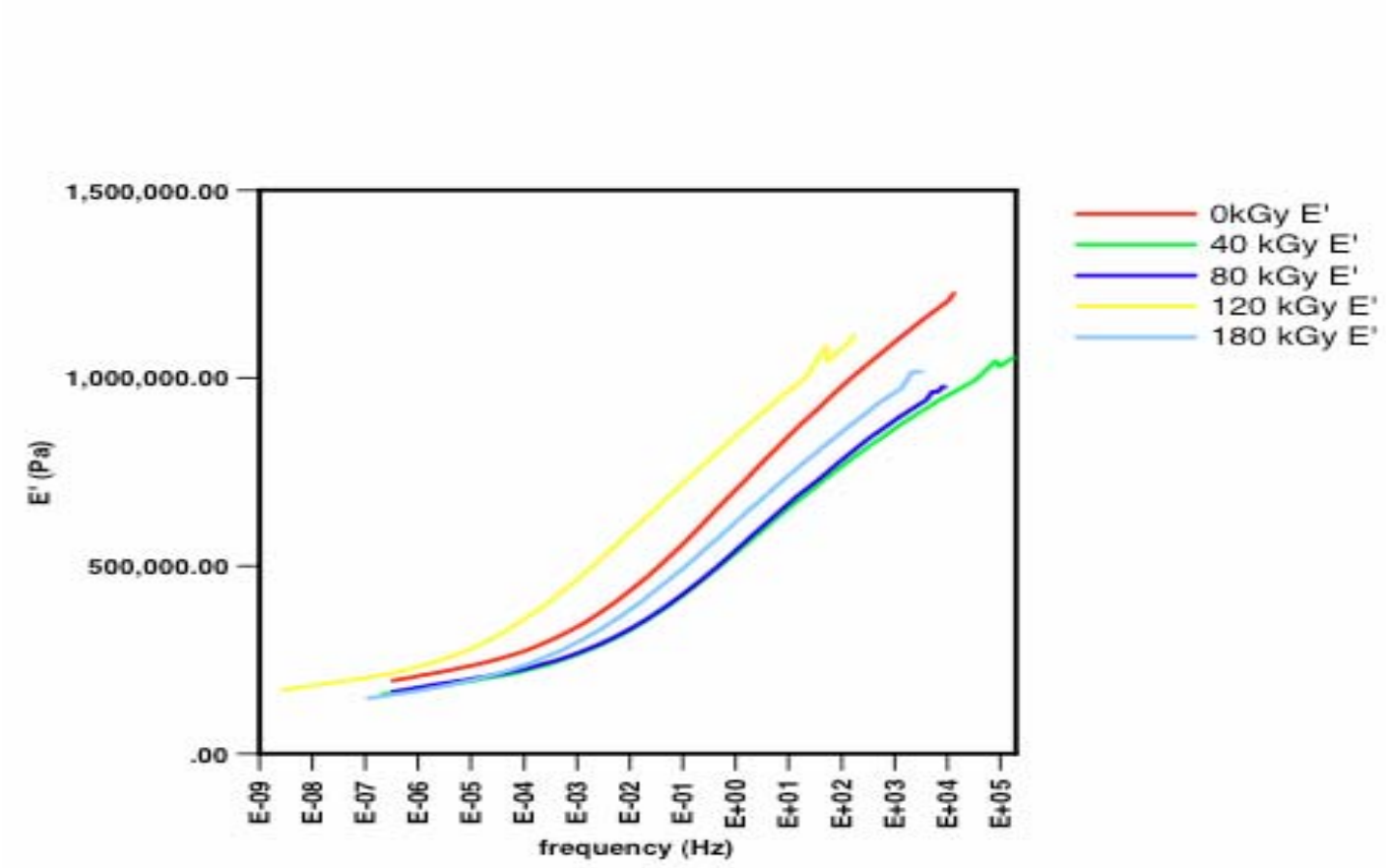

Figure 1: Storage modulus, E′ master curves for wood with varying e-beam dose levels.

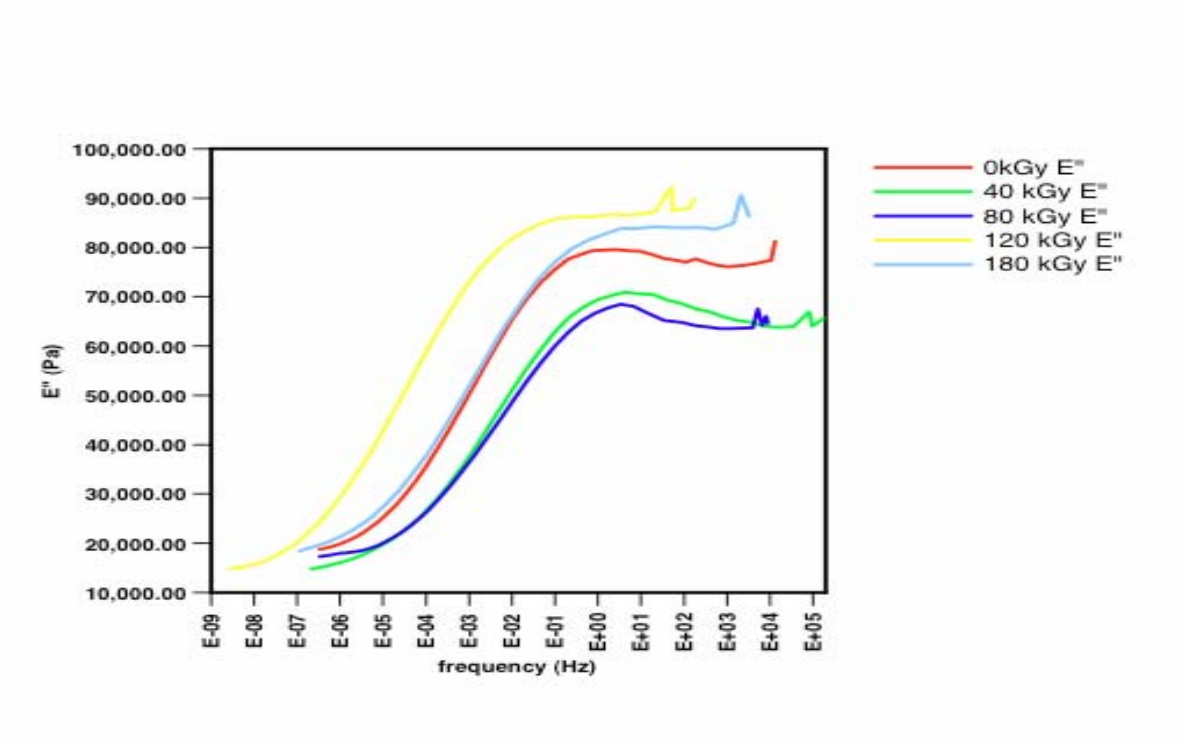

Figure 2: Loss modulus, E*"* master curves for wood with varying e-beam dose.

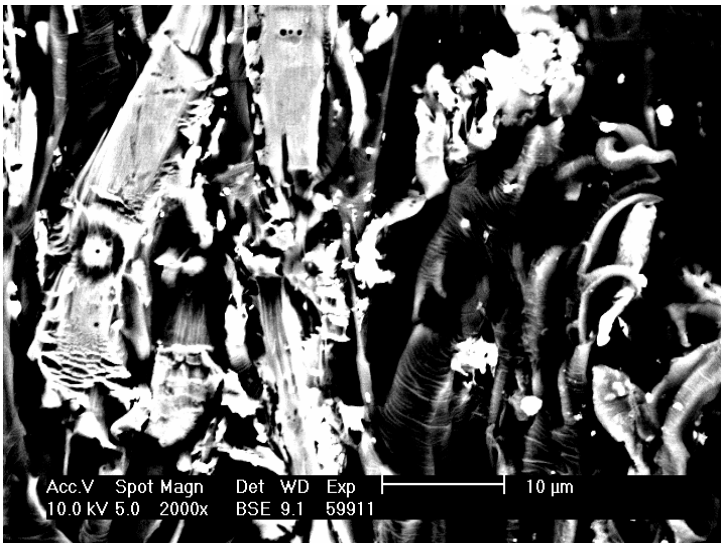

Figure 3: A backscatter image of a wood-polyethylene composite coupled with a zinc acrylate**.** The small white spots in the image are zinc particles confirmed by EDS.

Plans for Next Quarter: Evaluation of detailed dose-cure relationships will be continued. Additional wood composite samples and prototypes will be prepared for evaluation. Commercialization will continue to be evaluated. Planning for the fall technology transfer meeting will be continued. Work on the five-year report covering the entire project will continue. Several publications will be submitted for publication.

Patents: A disclosure covering the electron-beam curable additives for wood-plastic composites prepared

Publications/Presentations:

W. L. Griffith, 2006. DOE Industrial Technologies Forest Products Peer Review, April 5-6, 2006. Renaissance Waverly Hotel, Atlanta Ga .

W. L. Griffith, G. F. Dorsey, D. P. Harper, W. W. Moschler, Jr., T. G. Rials and Ting Song, P. M. Winistorfer, Chang Song, 2006. Electron-Beam Cured Resin Systems for Wood Composites, presented at ANTEC2006, May 6-11, 2006, Charlotte NC.

D. P. Harper, W. L. Griffith, T. G. Rials, G. Dorsey 2006. Electron Beam Processing of Wood-Polyethylene-Acrylic Composites To be presented at the SAMPE Long Beach Symposium. 16-20 May, Long Beach CA.

D. P. Harper, W. L. Griffith, T. G. Rials, G. Dorsey , K. Eugland, and M. P. Wolcott, Electron-Beam Curable Additives for Wood-Plastic Composites, Forest Products Society 60th International Convention , June 25-28, 2006, Newport Beach Marriott, Newport Beach, California, USA

Milestone Status Table:

ID Number	Task / Milestone Description	Planned Completion	Actual Completion	Comments
1.1	Survey existing wood adhesive systems	09/30/02	09/30/02	
1.2	Evaluation of initial resin systems	12/31/02	12/31/02	
1.3	Develop test protocol for block-tests	12/31/02	11/05/02	
1.4	Initiate adhesion studies	03/31/03	03/01/03	
1.5	Ethylenic and acetylinic bond systems	09/30/03	09/30/03	
1.6	Downselect promising resin systems	05/01/04	05/01/04	
1.7	Properties of glued wood assemblies	08/30/04	08/30/04	
1.8	Evaluation of process energy balance	09/30/04	09/30/04	
1.9	Initiate testing of large sections	01/01/05	12/23/04	
1.10	Evaluate alternative beam application methods including X-rays	09/30/05	09/30/05	
1.11	Select large-scale systems	01/01/06	01/01/06	
1.12	Technology transfer workshop	11/30/06		Rev. to increase attendance
1.13	Five-year Project Report	12/31/06		

Budget Data (as of 6/30/06):

			Approved Spending Plan			Actual Spent to Date		
Phase / Budget Period			DOE Amount	Cost Share	Total	DOE Amount	Cost Share	Total
	From	To						
Year 1[a]	10/01/01	9/30/02	200,000	71,000	271,000	116,000	65,000	181,000
Year 2	10/01/02	9/30/03	210,000	101,000	311,000	158,000	91,000	249,000
Year 3	10/01/03	9/30/04	220,000	101,000	321,000	262,000	91,000	353,000
Year 4[a]	10/01/04	9/30/05	230,000	101,000	331,000	21,000	91,000	112,000
Year 5[a]	10/01/05	9/30/06	240,000	71,000	240,000	59,000	54,000	123,000
Totals			1,000,000	445,000	1,445,000	616,000	392,000	1,008,000

[a]We received $150,000 in funding during FY 2002; $95,000 in FY2005; and $40,000 in FY2006.

Novel Isocyanate-Reactive Adhesives for Structural Wood-Based Composites

Frazier: Virginia Tech

GO14307

QUARTERLY PROGRESS REPORT

Project Title: Novel Isocyanate-Reactive Adhesives for Structural Wood-Based Composites

Covering Period: April 1, 2006 through June 30, 2006

Date of Report: 07/28/2006

Recipient: Virginia Polytechnic Institute and State University
Collegiate Square, 460 Turner Street, Suite 306
Blacksburg, VA 24060

Award Number: DE-FC36-04GO14307

Subcontractors: N A

Other Partners: National Starch and Chemical
TrusJoist - A Weyerhaeuser Business

Contact(s): Charles Frazier, 540 231 8318, cfrazier@vt.edu

Project Team: DOE: Beth Dwyer
Virginia Tech: Chip Frazier and Christian Heinemann
National Starch and Chemical: Charles Paul
TrusJoist: Kristin Brandt

Project Objective: The development of novel moisture-curing polyurethane adhesives for the cold-press manufacture of laminated veneer lumber made from high moisture content wood.

Background: Realizing the project objective, stated above, will reduce energy consumption through the elimination of hot pressing and through the substantial reduction of wood drying. Reduced wood drying will also curtail VOC production.

This is the 2nd quarterly project report in 2006 and 8th overall. Primarily, this report describes a new adhesive technology, delamination test results, and briefly the modified protocol for the laboratory-scale manufacture LVL boards.

Furthermore, a second method is addressed in order to determine rheological properties of bonded wood specimen.

Status:

a) Delamination Test (ASTM D2559-03) – National Starch and Chemical

The new emulsion polymer isocyanate (EPI) tested here is a two-part adhesive in which reactive latex is mixed with polymeric isocyanate (100:15). This adhesive which serves as the second adhesive system throughout the project was tested against the ASTM delamination test.

Several blocks were made for the adhesive using an add-on rate of 180 g/m^2 and a selected specific load of 150psi. The design of the block calls for orienting the substrates so that the growth rings are reversed at each layer, which focuses on bond strength in the z-direction. The test calls for the blocks to be soaked and then dried for a couple of cycles. As the wood picks up and subsequently looses moisture, it expands and contracts, based on the growth ring orientation. Because they are reversed at each layer, there is stress placed on the blocks at each glue line interface. Delamination is measured on the end grain surfaces only (both sides of the 5.0” wide portion). Since this is a hardwood, the allowable delamination percentage for the total block is 8%.

The results shown in Figure 1 reveal that the demanding test could be easily passed on Yellow-poplar (YP). So far, no data have been conducted for the EPI on Southern Yellow Pine (SYP).

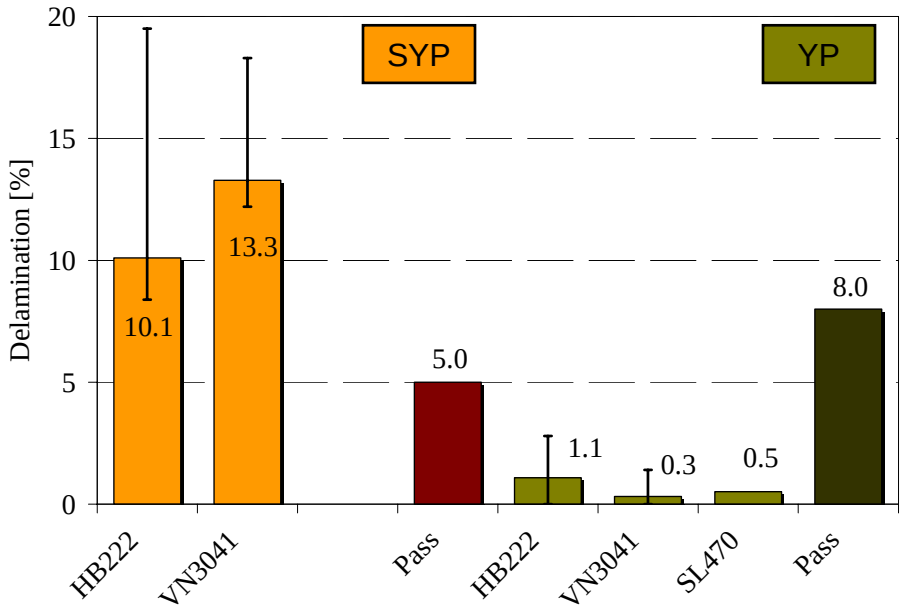

Figure 1: Delamination test results (ASTM D2559-03) of different adhesive systems on Southern Yellow Pine and Yellow-poplar. The new Latex-pMDI blend is named SL470.

b) Small-Scale Manufacture of LVL – Virginia Tech –

The new adhesive technology for small-scale manufacture of LVL is a two-part reactive latex (SL470) which is crosslinked by polyisocyanates. The adhesive is a product of the industrial project collaborator National Starch and Chemical (NSC). Before manufacturing small-scale LVL a preliminary billet with four veneer layers was pressed. This was necessary in order to get used to the handling of the new adhesive technology and introduce the method to new students helping with the lay-up procedure.

The applied mixing procedure was coordinated with our project partner NSC. On 100 parts of the latex 15 parts adhesive were added. For the Southern Yellow Pine billets, assembled with 12 veneer layers, a total of 805 g adhesive was mixed for 10 min in a 1000 ml beaker. A stirring propeller with 4 blades and of 88.5 mm in diameter was used at speed of 200 rpm for the mixing. The adhesive amount for the Yellow-poplar billets assembled of 10 veneer layers was 667 g keeping the same mixing parameters as for the SYP billets.

For this second batch of lab-scale LVL with the new emulsion polymer isocyanate (EPI) adhesive technology the established lay-up procedure from the previous batch was applied which is described in detail in the quarterly report 3_Q1_2005. Just minor changes were made regarding the application of the adhesive. In the first batch of LVL manufacturing it was found that it was easier to spread the adhesive on the veneer's "tight" side, rather then on the "loose" side containing laith checks. In this series of LVL manufacturing the latex – isocyanate blend was applied onto the "tight" side of the veneers only before the billet was assembled symmetrically with respect to the “loose” and the “tight” veneer surfaces. One exception was necessary for the center bond line where two "loose"-sides where bonded together. The recommended add-on rate for this adhesive was 180 g/m^2 which corresponds a mass of 66.9 g per bond line. An additional amount of 3 g were added to each center bond line due to the fact that two "loose" veneers sides where bonded together.

The press was operated manually using a specific pressing load of 1 N/mm^2 as the controlled process variable. During the recommended pressing time of 2 hours to achieve total cure the billets lost moisture. Just after press opening the weight and the thickness in 4 positions of each of the pressed billets were taken before they were stored in the conditioning chamber until being shipped to Trus Joist for mechanical testing.

c) Thermo gravimetric Analysis (TGA)

With the thermo gravimetric analysis (TGA) the point of adhesive degradation is determined. This information is important for all subsequent rheology experiments to avoid any degradation when using these very sensitive instruments. The degradation of neat latex and of cured latex-isocyanate was found to start at the same temperature of 220°C (Figure 2).

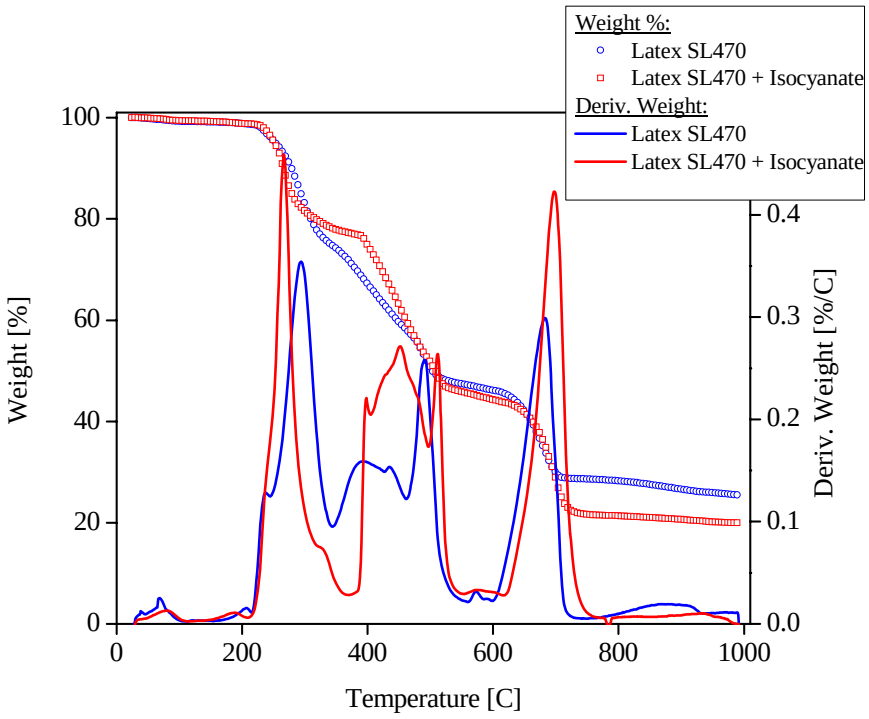

Figure 2: TGA of neat latex and additional iscocyanate (100:15)

d) Viscosity measurement and cure study

The viscosity of the neat latex was determined at 25°C using the cone-and-plate geometry with 40 mm in diameter and 2 degrees angle on an AR1000 rheometer. In a preliminary continuous ramp step experiment the optimum settings for the continuous flow experiment were determined. A total of 4 runs were conducted, two from high to low torque, and two in the reverse order. The results shown in Figure 3 reveal the shear thinning properties of the latex. When comparing the viscosity results at lower shear stresses, it becomes obvious that the experiments conducted from high to low torque shows a reduced viscosity values than those determined in the reverse order. This might be due to the fact that the latex structure is already affected at high shear stresses and polymeric linkages are broken up.

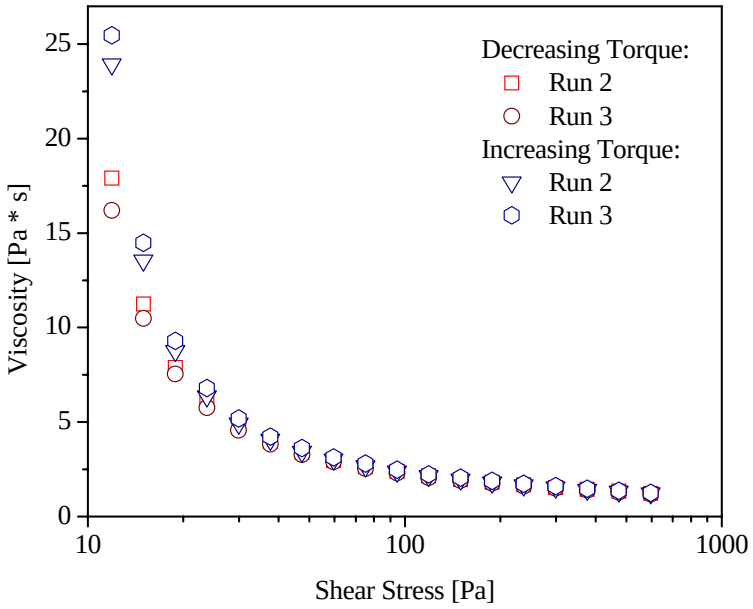

Figure 3: Continuous flow experiment conducted from high to low and from low to high torque.

In another test the isothermal cure of the latex-isocyanate blend was determined using disposable aluminum plates of 25 mm in diameter. A volume of 0.61 ml was cured at 25°C and complete cure was reached after approximately 240 min. The gelation time was found to be close to 60 min (Figure 4) indicated by the tan(delta) peak.

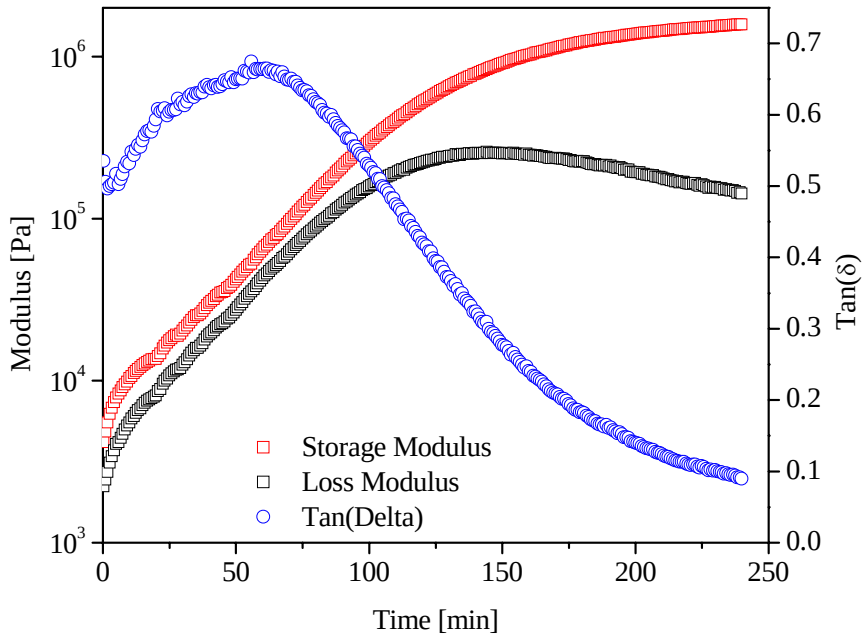

Figure 4: Curing reaction of latex-isocyanate blend at an isothermal temperature of 25°C.

e) Rheology measurements of bonded wood samples in torsion mode – Virginia Tech

In a new series of experiments laminated wood samples, bonded with the 100% organic polyurethane adhesive, were tested in torsion mode using the AR2000 rheometer. This second test series is necessary since the previous experiments with the parallel plate geometry did not reveal any difference regarding the shift factor plot derived from frequency sweep experiments of weathered and unweathered specimens although the adhesive failed the delamination test (ASTM D2559-03). This standard is a weathering procedure under severe conditions of steam and temperature. Two significant differences were encountered with the new torsion geometry and related deformation mode. First, the sample's design corresponds more to a realistic adhesive cure of laminated wood composites with closed bond lines and wood layers on the outside. Second, the torsion mode was chosen to reveal differences in the response of weathered and unweathered samples and help understanding why the adhesive is not passing the required delamination test on SYP.

The sample preparation, lay-up and pressing procedure applied here, was similar to the one used for the specimens tested in parallel plate configuration. However, the significant difference of the new sample was related to complete cure of the adhesive in between two layers of wood. In contrast to the sample preparation of the parallel plate samples which had been disassembled after 4 min even before gelation was achieved.

Southern Yellow Pine and Yellow-poplar specimens were cut from conditioned (20°C and 65% RH) wooden blocks to dimensions of 280 mm in length and 55 mm in width after being planed to 1.3 mm in thickness. A total of six 3-ply laminated samples were manufactured with an adhesive spread rate of 170 g/m^2. A specific pressing load of 0.001 N/mm^2 was applied to the samples. After a pressing time of 90 min the specimens were stored in a conditioning chamber until total cure was reached. Then the samples were cut to their final dimensions of 35 mm in length and 10 mm in width, featuring two different grain orientations, longitudinal and radial. Prior to testing the samples were dried in a desiccator over P_2O_5 and under Nitrogen atmosphere.

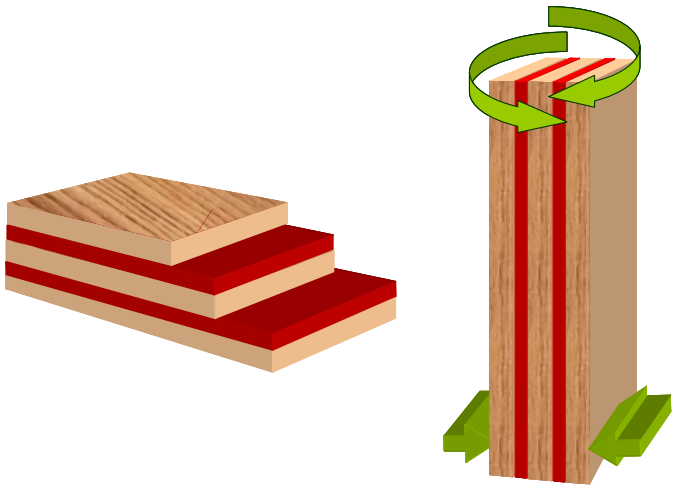

Figure 5: Left: 3-layered laminated sample bonded with polyurethane adhesive (red). Right: Sample in its actual torsional deformation mode.

Determination of the linear viscoelastic region (LVR)

The linear viscoelastic region of a laminated SYP sample was determined in a stress sweep experiment. The length of the sample featured the radial orientation of the wood layers clamped at its ends within torsion set-up of the instrument. In Figure 6 the results of the LVR experiment are shown at 3 different temperatures. The decision for the LVR is based upon the 95% criteria of the maximum storage modulus value. With increasing oscillational stress, the signal quality increases. For any further experiments the highest oscillational stress possible was applied. Since this is a function of temperature a value of $4.0*10^4$ was determined for 180°C and $4.0*10^5$ was found to be appropriate for - 60°C.

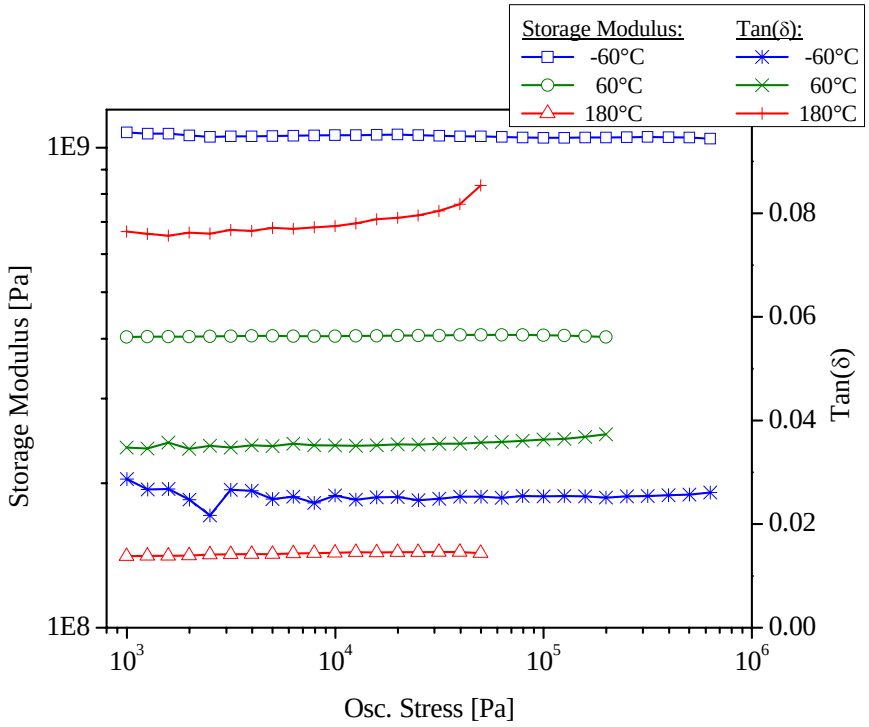

Figure 6: Storage modulus and tan(delta) response from LVR measurement for SYP radial samples.

Thermal Scans

In the first approach thermal scans were conducted using the bonded wood samples. During the first heating scan the (thermal) history of the sample was erased and the polymeric adhesive becomes more monolithic. In the subsequent thermal scan, here conducted in cooling, or any following experiment the sample shows a distinct difference compared to the first thermal scan. In Figure 7 the response of a laminated wood sample during an initial thermal scan and the subsequent cooling scan is shown. The storage modulus and the tan(delta) curves feature a major transition from 40°C to 60°C in the first thermal scan. After a conditioning period of 15 min at 180°C a totally different response was observed for the subsequent thermal scan (cooling). Especially,

the particular transition from the first thermal scan did not occur again. Instead, a minor transition was found in the high temperature region and a large transition could be detected between 0°C and -40°C before the glassy plateau was reached.

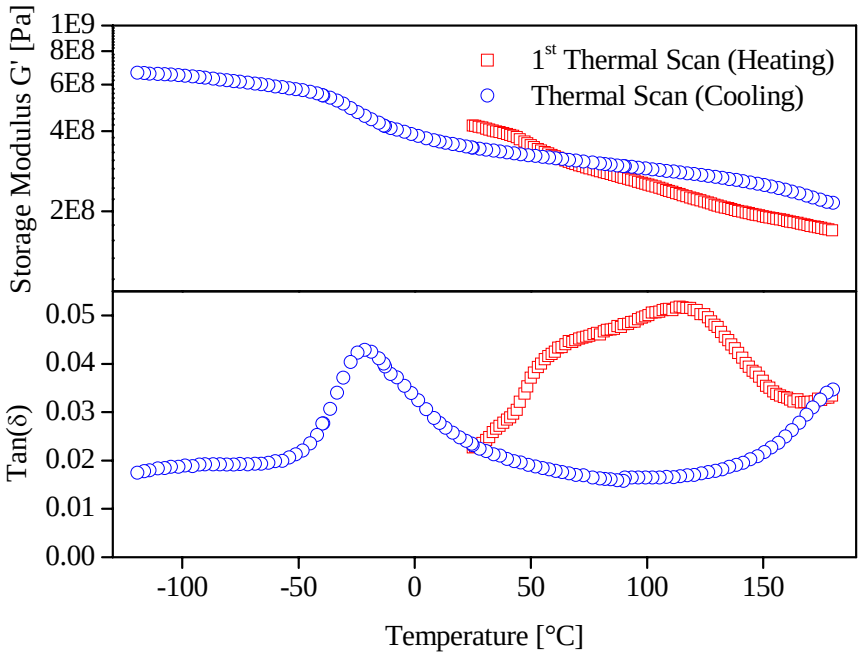

Figure 7: Storage Modulus and tan(delta) curves obtained from 1st and 2nd thermal scans.

Comparing the results obtained from parallel plate and the torsion experiments, differences over the measured temperature window are evident. Addressing the elastic properties of the sample it was found, that the storage modulus of the torsion experiment was higher than the one determined in the parallel plate experiment. Furthermore, the change in modulus of the torsion samples spans less than half a decade within the observed temperature window, whereas the parallel plate sample spans 1.5 decades. This observation can be explained by the test mode. In the parallel plate experiment torsion forces mainly occur within the bond lines while in pure torsion of a solid sample the response of the wood surface layers is predominant.

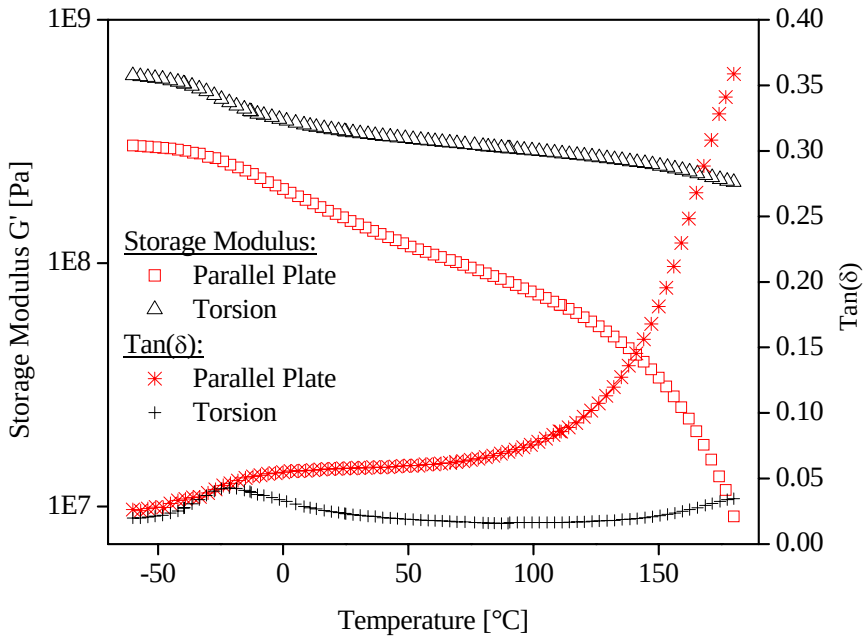

Figure 8: Comparison of thermal scan after conditioning at 180°C of different geometries and deformation modes.

Frequency Sweep Experiments

In order to obtain frequency mastercurves and shift factors for the new geometry and deformation mode isothermal frequency sweep steps had to be conducted from -60°C to 180°C. In numerous preliminary tests optimal settings had to be found in order to achieve the required data quality for the mastercurves. Longitudinal Yellow-poplar samples of 50 mm in length were chosen to serve as specimens. It was found that the storage modulus of the isothermal frequency steps could be shifted along the frequency axis until they coincide, whereas difficulties were encountered with the tan(delta) values. Isothermal frequency steps hardly coincide within a temperature window from 20°C to 120°C when curves were shifted by the obtained storage modulus values. The discontinuity occurred at lower frequencies. Neither adjusting the various instrument parameters nor changes in sample dimensions in order to allow more compliance have solved the inconsistency of the tan(delta) mastercurves.

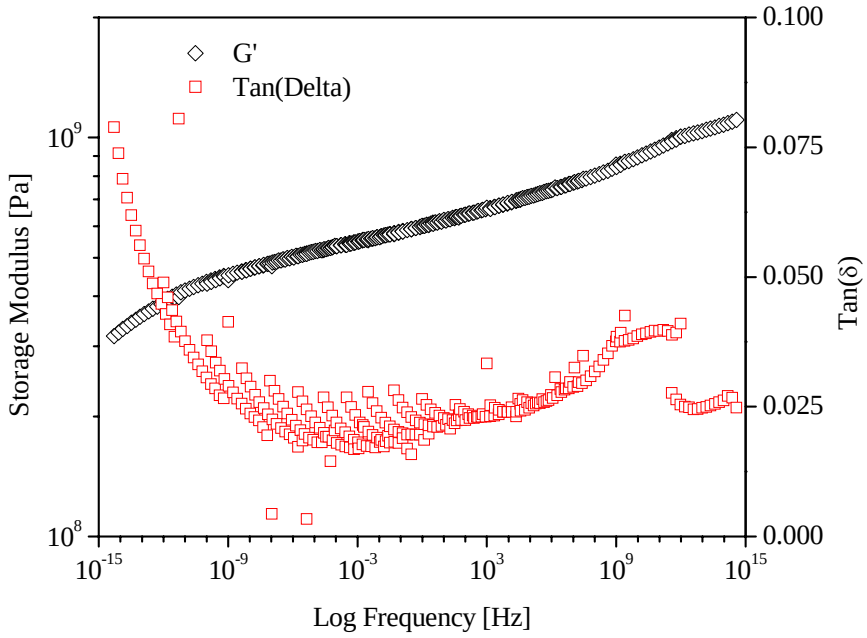

Figure 9: Storage modulus and tan(delta) master curves obtained from isothermal frequency experiments.

Better results were obtained with a new set of bonded wood samples using Southern Yellow Pine with cross grain (radial) orientation. The data shown in Figure 10 represent the results within the critical temperature range from 20°C to 120°C. The shift of the storage modulus of the isothermal frequency steps resulted in a smooth mastercurve. However, the tan(δ) shift was not perfectly aligned but it has definitely improved compared to the those samples with longitudinal orientation. The fact that the tan(δ) shift still shows some inconsistency might lead to the conclusion that the selected deformation mode with bonded wood samples is questionable if the time temperature superposition (TTS) principle is applicable. Nevertheless, the new sample was encouraging. More tests have to be performed to find the optimal settings of the instrument and sample geometry.

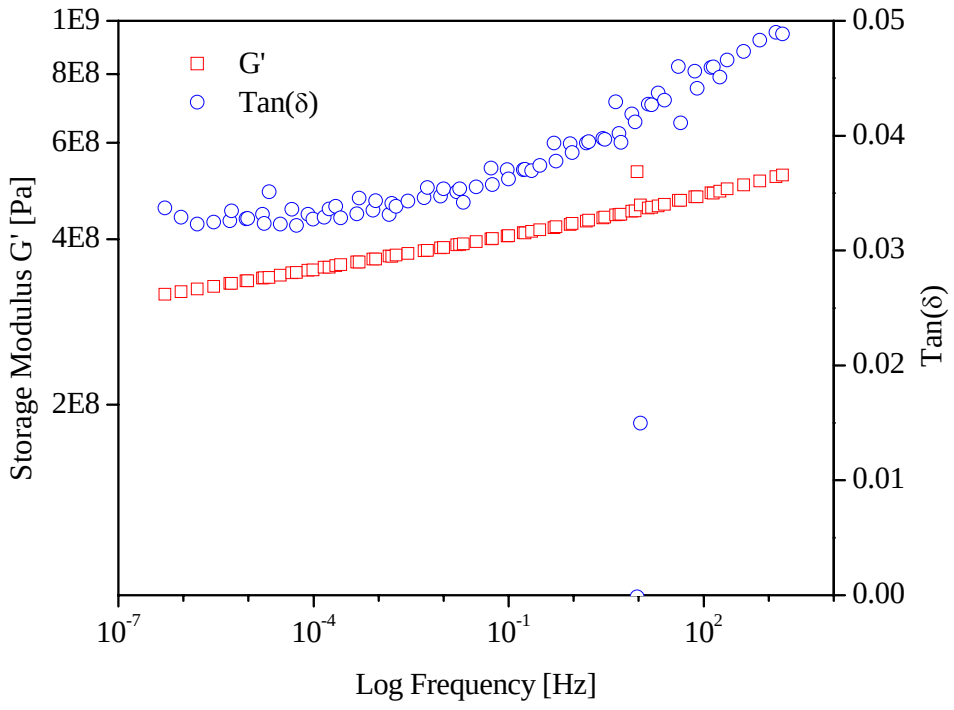

Figure 10: Storage modulus and tan(delta) mastercurve obtained from isothermal frequency measurements.

Milestone Status Table as of September 13, 2005:

ID Number	Task / Milestone Description	Planned Completion	Actual Completion	Comments
1.1.1	VT: Install hydraulic fluid cooling system on pilot-scale hot-press	Jan 05	Jan 05	
1.1.2	VT: Install new Rheometer system	Dec 04	Jan 05	
1.1.3	VT: Develop small-scale LVL manufacture protocol.	Feb 05	Jan 05	
1.1.4	NS: Install autoclave for ASTM D2559 testing	Dec 04	Feb 05	
1.1.5	NS: Initiate ASTM D2559 testing for phase I adhesives	Jan 05	March 05	
1.2.1	VT: Install RH control system	Jan 05	Feb 05	
1.2.2	VT: Initiate development of isothermal /iso-humid cure analysis	Feb 05	March 05	
1.2.3	VT: Manufacture SYP-LVL	Feb 05	June 05	
1.2.4	VT: Manufacture SP-LVL	March 05	June 05	
1.2.5	NS: Complete ASTM D2559 testing	Feb 05	June 05	
1.2.6	NS: Conduct adhesive thermal, swelling etc. analysis	Jan 05	May 05	
1.2.7	TJ: Initiate small-scale LVL-testing	Feb 05	March 05	
1.3.1	VT: Complete development of isothermal/iso-humid rheometer cure analysis.	April 05	May 05	
1.3.2	VT: Initiate crosslink density (Rheometer) measurements of neat phase I adhesives in parallel plate mode.	May 05	Sept 05	
1.3.3	VT: Initiate development of crosslink density measurements of bonded-wood samples in parallel plate mode	June 05	Oct 05	
1.3.4	NS: Compile and review phase I results.	June 05	June 05	
1.3.5	NS: Devise phase II adhesive synthesis/formulation strategy.	June 05	July 05	
1.3.6	TJ: Complete phase I small-scale LVL mechanical testing.	May 05	July 05	
1.4.1	NS: Prepare phase II adhesives.	Aug 05	Aug 05	
1.4.2	NS: Conduct ASTM D2559 testing for phase II adhesives.	Sept 05	Sept 05	
1.4.3	NS: Conduct adhesive thermal, swelling, pot-life, etc. analyses.	Sept 05	Sept 05	
1.4.4	VT: Manufacture southern pine LVL; 6 billets for phase II adhesive (+2 billets for PF control).	Sept 05	May 06	
1.4.5	VT: Manufacture yellow-poplar LVL; 6 billets for phase II adhesive (+2 billets for PF control).	Sept 05	May 06	
Year 2:				
2.1.1	TJ: Conduct phase II small-scale LVL mechanical testing.	Dec 05		June 06

ID Number	Task / Milestone Description	Planned Completion	Actual Completion	Comments
2.1.2	VT: Conduct phase II adhesive cure & crosslink density studies.	Dec 05		June 06
2.1.3	NS: Compile and review phase II results.	Nov 05		June 06
2.1.4	NS: Devise phase III adhesive synthesis/formulation strategy	Dec 05		July 06
2.1.5	NS: Organize phone conference (**Milestone #1**)	Dec 05	Sept 05	Project group meeting held in Blacksburg, VT
2.2.1	NS: Prepare phase III adhesives	Jan 06		August 06
2.2.2	NS: Conduct ASTM D2559 testing for phase III adhesives	Feb 06		September 06
2.2.3	NS: Conduct adhesive thermal, swelling, pot-life, etc. analyses	March 06		September 06
2.2.4	VT: Manufacture southern pine LVL; 6 billets for phase III adhesives (+2 billets for PF control)	March 06		September 06
2.2.5	VT: Manufacture yellow-poplar LVL. 6 billets for phase III adhesives (+2 billets for PF control).	March 06		September 06
2.3.1	TJ: Conduct phase III small-scale LVL mechanical testing.	June 06		October 06
2.3.2	VT: Conduct phase III adhesive DMA cure & crosslink density studies.	May 06		October 06
2.3.3	NS: Compile and review phase III results.	May 06		November 06
2.3.4	NS: Devise phase IV adhesive synthesis/formulation strategy.	June 06		December 06
2.3.5	NS: Organize phone conference (**Milestone #2**).	June 06		September 06
2.4.1	NS: Prepare phase IV adhesive.	July 06		
2.4.2	NS: Conduct ASTM D2559 testing for phase IV adhesives.	Aug 06		
2.4.3	NS: Conduct adhesive thermal, swelling, pot-life, etc. analyses.	Sept 06		
2.4.4	VT: Manufacture southern pine LVL; 6 billets for phase IV adhesive (+2 billets for PF control).	Sept 06		
2.4.5	VT: Manufacture yellow-poplar LVL; 6 billets for phase IV adhesive (+2 billets for PF control).	Sept 06		
Year 3:				
3.1.1	TJ: Conduct phase IV small-scale LVL mechanical testing.	Nov 06		
3.1.2	VT: Conduct phase IV adhesive cure & crosslink density studies.	Nov 06		
3.1.3	NS: Compile and review phase IV results.	Nov 06		
3.1.4	NS: Devise phase V adhesive synthesis/formulation strategy.	Dec 06		
3.1.5	NS: Organize research review at TJ Technology Center, Boise ID (**Milestone #3**).	Dec 06		
3.2.1	NS: Prepare phase V adhesives.	Jan 07		

ID Number	Task / Milestone Description	Planned Completion	Actual Completion	Comments
3.2.2	NS: Conduct ASTM D2559 testing for phase V adhesives	Feb 07		
3.2.3	NS: Conduct adhesive thermal, swelling, pot-life, etc. analyses.	March 07		
3.2.4	TJ: Manufacture large-scale southern pine LVL; 2 billets for phase V adhesive (+2 billets for PF control)	Feb 07		
3.2.5	TJ: Manufacture large-scale yellow poplar LVL; 2 billets for phase V adhesive (+2 billets for PF control)	Feb 07		
3.2.6	TJ: Conduct phase V large-scale LVL mechanical testing	March 07		
3.2.7	TJ: Analyze mill trial feasibility.	March 07		
3.2.8	VT: Conduct phase V adhesive cure & crosslink density studies.	March 07		
3.3.1	NS: Compile and review phase V results.	April 07		
3.3.2	NS: Devise phase VI adhesive synthesis/formulation strategy.	April 07		
3.3.3	NS: Organize phone conference (**Milestone #4**).	June 07		
3.3.4	NS: Prepare phase VI adhesive.	May 07		
3.3.5	TJ: Manufacture large-scale southern pine LVL; 2 billets for phase VI adhesives (+2 billets for PF control).	May 07		
3.3.6	TJ: Manufacture large-scale yellow-poplar LVL; 2 billets for phase VI adhesives (+2 billets for PF control).	May 07		
3.3.7	TJ: Conduct phase VI large-scale LVL mechanical testing	June 07		
3.3.8	VT: Conduct phase VI adhesive cure & crosslink density studies.	June07		
3.4.1	NS: Conduct ASTM D2559 testing for phase VI adhesive	July 07		
3.4.2	NS: Conduct adhesive thermal, swelling, pot-life, etc. analyses.	Aug 07		
3.4.3	NS: Compile and review phase VI results.	Sept 07		
3.4.4	NS: Organize research review and planning session (**Milestone #5**).	Sept 07		

Project Spending and Estimate of Future Spending							
Quarter	**From**	**To**	**Estimated Federal Share of Outlays***	**Actual Federal Share of Outlays**	**Estimated Recipient Share of Outlays***	**Actual Recipient Share of Outlays**	**Cumulative**
4Q04	Start(10/15/04)	12/31/04	$98,000	$40,216.50	$18,240.75	$1,395.00	0
1Q05	1/1/05	3/31/05	$25,000	$116,993.83	$18,240.75	$2,894.00	
2Q05	4/1/05	6/30/05	$25,000	$19,421.99	$18,240.75	$18,736.00	
3Q05	7/31/05	9/30/05	$30,578	$24,690.00	$18,240.75	$29,142.25	
4Q05	10/1/05	12/31/05	$25,000	$19,643.15	$22,990.75	$15,364.00	
1Q06	1/1/06	3/31/06	$36,671.63	$19,151.00	$22,990.75	$8,445.00	
2Q06	4/1/06	6/30/06	$36,671.63	$25,834.15	$22,990.75	$8,612.00	
3Q06	7/1/06	9/30/06	$36,671.63		$22,990.75		
4Q06	10/1/06	12/31/06	$36,671.63		$14,674.25		
1Q07	1/1/07	3/31/07	$36,671.63		$14,674.25		
2Q07	4/1/07	6/30/07	$36,671.63		$14,674.25		
3Q07	7/1/07	9/30/07	$36,671.63		$14,674.25		
4Q07	10/1/07	12/31/07	$24,351.63				
Totals			**$479,053**	$265,950.62	$223,623	$84,588.65	**0**

Biological Air Emissions Control for an Energy Efficient Forest Products Industry of the Future

Jones: Texas A&M

GO14310

QUARTERLY PROGRESS REPORT

Project Title: **Biological Air Emissions Control for an Energy Efficient Forest Products Industry of the Future**

Covering Period: **April 1, 2006 through June 30, 2006**

Date of Report: **July 31, 2006**

Recipient: **South Texas Environmental Institute**
Texas A&M University-Kingsville
Department of Environmental Engineering, MSC 213
Kingsville, Texas 78363

Award Number: **DE-FC36-04GO14310**

Subcontractors: **Bio•Reaction Industries LLC**
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Other Partners: **Stimson Lumber Company**
Forest Grove, OR

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Project Objectives

The gaseous emissions from the hardboard mill presses at lumber plants such as that of the Stimson Lumber Company contain both volatile and condensable organic compounds (VOC and COC, respectively), as well as fine wood and other very small particulate material. In applying bio-oxidation technology to these emissions Texas A&M University-Kingsville (TAMUK) and BioReaction (BRI) will address and evaluate the potential of this equipment to resolve two (2) control issues which are critical to the industry:

- First, the hazardous air pollutant (HAP) emissions (primarily methanol and formaldehyde) and
- Second, the fine particulate and COC from the press exhaust which contribute to visual emissions (opacity) from the stack.

To accomplish these objectives TAMUK and BRI are evaluating a biological treatment technology that will demonstrate significantly lower energy use (than regenerative thermal oxidizers (RTOs) or regenerative catalytic oxidizers (RCOs), lower water use (than conventional scrubbers) all the while being less costly than either for maintenance.

Q2 2006 Status

The evaluation of the field scale biological treatment test evaluation for the unit at Stimson Lumber Company at Forest Grove, Oregon was completed in April 2006. The unit collected and treated emissions from the pressboard steam vents on the top of the plant building and delivered the emissions into the biological control unit consisting of a biotrickling unit and a biomatrix-filled biofilter module in series, with air flow capacity of up to 10,000 cfm. This unit successfully treated air flow emissions ranging from approximately 3,500 to 1,500 cfm during the testing phases, and provided an EBRT (empty bed residence time) of only 45 seconds. FID gas measurements from the unit indicated high removals of total VOCs coming from the press with a reduction down to less than 5 ppm at the outlet of the biological system. Evaluations of opacity reductions have also met project objectives with routine outlet measurements from 0-5% opacity readings, which were in compliance with regulatory guidelines. A scale up of the biological treatment technology to handle all of Stimson's press vent emissions output of almost 75,000 cfm is feasible.

Water quality parameters, such as conductivity and pH, were measured in the trickling unit sump and correlated to system performance. Nutrients in samples collected from the unit system's sump were also tested and analyzed for nitrates, sulfates and chemical oxygen demand. Solid media samples were also collected periodically for tests on nutrients and microbial activity. The results of these tests demonstrated that the fresh water steam condensation from the press vent kept the water quality in the trickling filter at acceptable levels without generating large quantities of discharge water. Also, the solid samples showed extensive protein and biological based materials demonstrating significant biofilm development in the biofilter media.

In the first quarter of 2006, there was a plant shutdown due to scheduled process maintenance. A FID test was completed before and after this shutdown period which

proved that bio-oxidation unit performance was not affected for this three week period of inactivity.

Emissions samples were also collected at different points in the biological system with the use of silica lined canisters. These canisters were shipped to the Texas A&M University-Kingsville Environmental Engineering laboratories for Gas Chromatography-Mass Spectrometry (GC-MS) analysis to determine and measure more volatile organic compounds which could contribute to the opacity emissions. Significant removal of alpha-pinene and other aldehyde based emissions were measured in the canister samples.

On the technology commercialization side, BioReaction Industries (BRI) has been actively working with the Wood Products industry over the past year. In May, a three-month pilot evaluation was concluded very successfully at a major wood products company OSB mill (processing southern yellow pine) for a press vent. A slipstream off the press emissions exhaust was treated in the BRI bio-oxidation system, with VOC removal efficiencies exceeding 75%. These data provided additional support for a determination by the state and USEPA Region IV that the BRI bio-oxidation system was acceptable as best available control technology (BACT) for press emissions. BRI is in final negotiations for a press emissions bio-oxidation system. Additionally, BRI is working with three other Wood Product companies toward the purchase and installation of bio-oxidation systems to control press emissions from several panel board mills.

Q3 2006 Plans

To further enhance the technology's commercialization and reduce technology startup costs, allowing for broader implementation by the forest products industry, additional field testing and laboratory testing will be needed. This testing should be focused on developing increased optimization parameter criteria and performance evaluation before the implementation of MACT guidelines by EPA, which were postponed until mid 2008.

Additional funding is being requested to continue this research on both the bench and pilot scales to decrease unit retention time and yet still achieve MACT standards, thereby reducing the field scale unit footprint, size and capital cost for forest products plants.

Patents: None this Quarter

Publications/Presentations:

Santos, S., Jones, K., Baliwala, L., Abdul, R., Boswell, J. and Cochran, J. (2006) Treatment of Wet Process Hardboard Plant Emissions by a Pilot Scale Biological System, submitted to the 2006 USC-TRG Conference on Biofiltration for Air Pollution Control, Long Beach, California, October 18-20.

Jones, K., Santos, S., Baliwala, L., Boswell, J. and Paca, J. (2006) Biological air emissions control for α-pinene and formaldehyde for a forest product industry application with a coupled biotrickling filter and biofilter system, in Proceedings of 17th International Congress of Chemical and Process Engineering CHISA 2006, Prague, Czech Republic, 27–31 August 2006.

Jones, K., Boswell, J., Santos, S., Cochran, J., and John, P. (2006) Field Scale Evaluation of a Combined Biotrickling Filter/Biofiltration Unit for Treatment of Pressboard Manufacturing Emissions, in preparation for submission to the *Journal of Clean Technologies and Environmental Policy*.

Santos, S., Jones, K., and Boswell, J. (2005) "Biological Treatment of Air Emissions for Forest Product Industry Applications," presented at the NSF sponsored CREST-RESSACA Conference on Emerging Technologies for a Sustainable Environment, October 20-21, South Padre Island, Texas.

Milestone Status Table:

ID No.	Tasks/Milestones	Planned Completion	Actual Completion	Comments	
1.	Bench Scale and Field Scale Unit Design	12/31/04	12/31/04		
2.	Field Pilot Unit Construction	6/30/05	8/15/05		
3.	Field Pilot Unit Startup	7/31/05	8/15/05		
4.	Preliminary Pilot Scale Testing and Field Testing Plan Development	10/1/05	8/15/05		
5.	Optimization of Biotrickling Filter	12/31/05	3/15/06		
6.	Optimization of Biofilter Section	12/31/05	3/15/06		
7.	Develop process models	2/28/06		In development	
8.	Optimize water reuse	2/28/06	3/15/06		
9.	Characterize microbial biofilm	2/28/06	6/15/06		
10.	BF Tech Product marketing	8/31/06		Ongoing	
11.	Final Report/Publication	8/31/06		In preparation	

Budget Data:

Texas A&M University – Kingsville Award DE-FC36-04GO14310 **Project Spending and Estimate of Future Spending**							
Quarter	**From**	**To**	**Estimated Federal Share of Outlays***	**Actual Federal Share of Outlays**	**Estimated Recipient Share of Outlays***	**Actual Recipient Share of Outlays****	**Cumulative**
	Start		Note 1		Note 1		
3Q04	9/1/04	9/30/04	0	0	0	0	
4Q04	10/1/04	12/31/04	4,642	4,642	2,000	2,000	
1Q05	1/1/05	3/31/05	3,922	3,922	3,500	3,500	
2Q05	4/1/05	6/30/05	18,955	18,955	15,000	15,000	
3Q05	7/31/05	9/30/05	25,649	25,649	20,000	20,000	
4Q05	10/1/05	12/31/05	26,696	26,696	17,265	17,265	
1Q06	1/1/06	3/31/06	95,220	95,220	34,235	34,235	
2Q06	4/1/06	6/30/06	15,892	15,892			
3Q06	7/31/06	9/30/06	1,753	1,753			
4Q06	10/1/06	12/31/06					
1Q07	1/1/07	3/31/07					
2Q07	4/1/07	6/30/07					
Totals			**192,729**		**92,000**	**92,000**	

* Update quarterly
**Estimated for 2006
General Note: DOE Laboratory partner spending should not be included in the above table. If a DOE Laboratory is a partner, report their spending and spend plan information in the table below (use separate tables if multiple DOE Laboratories are involved).
General Note: The information in this table should be consistent with the information provided in section 10 of the quarterly financial status reports (SF269 or SF269A).
Note 1: Leave blank. Only the actual DOE/Cost Share amounts spent through 6/30/04 are needed.
Note 2: Amount for this quarter and subsequent quarters should be updated as necessary on a quarterly basis. Estimates need to be provided for the entire project. If spending for a given quarter is different than estimated, then the remaining quarter's estimates should be updated to account for the difference. Total DOE and Cost Share amounts should be the same as the Award amount.

On-Line Oxidation of Volatile Compounds Generated by Sawmill Wood Kilns

Kumar: Mississippi State University

GO14311

QUARTERLY PROGRESS REPORT

Project Title: On-line Oxidation of Volatile Organic Compounds Generated by Sawmill Wood Kilns

Covering Period: April 1, 2006 through June 30, 2006

Date of Report: July 28, 2006

Recipient: Mississippi State University: Diagnostic Instrumentation and Analysis Laboratory (DIAL) and the Department of Forest Products (FP)
Award Number: DE-FC36-04GO14311

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Project Objective: The objective of this project is to evaluate the effectiveness of the use of hydrogen peroxide in combination with ultra-violet (UV) light for the reduction in the amount of volatile organic compounds (VOCs) released into the environment during wood drying in sawmill kilns.

Background: About thirty thousand short tons of VOCs are released into the environment during kiln drying of softwood species, in particular southern yellow pine. The traditional methods for controlling VOC emission, such as regenerative thermal oxidizers (RTO), recuperative catalytic oxidizers (RCO), or bioreactors are either expensive or very difficult to apply due to the large variations in humidity and VOC flow that occur during the drying process. In particular, the humidity fluctuations reduce the effectiveness of oxidation-type control devices and are detrimental to the organisms in bioreactors which are also affected by temperature variations and interruptions in VOC output.

The technique evaluated in our work is a form of chemical oxidation referred to as an "Advanced Oxidation Process" (AOP). In this technique, UV photolysis of hydrogen peroxide results in the formation of hydroxyl radicals that oxidize or break down the organic pollutants contained in the kiln emissions. Preliminary testing under UV light indicated that a VOC reduction as high as 40% could be obtained. This testing was conducted at MSU by flowing kiln off gas through a weak hydrogen peroxide solution. It was estimated that a much higher reduction could be obtained through optimization of the design of the reaction cell and the process parameters.

Status: A thorough reevaluation of the principles determining the design of the reaction cell was conducted. The new reaction cell is a combination of a mini packed column, to knock out some particulates, followed by the VOC treatment chamber. A schematic of the cell is shown in Fig. 1. VOC laden air enters the bottom of the lower chamber from the right side. It flows up through a layer of marbles into the upper treatment section. The marbles provide the dual function of distributing the air through the cross-section, as well as providing for an efficient liquid/gas interface with the re-circulating peroxide solution to knockout some of the particulates. The gas then enters the treatment section which is currently a chamber 24" tall and 18" in diameter. A quartz tube runs across the middle of the chamber which houses the UV lamp(s) that aid in the treatment of the VOCs. The treated gas then exits the top of the chamber. Gas sampling probes are located at the inlet and outlet of the test chamber in order to evaluate the VOC removal efficiency. The total height of test cell is 50". Peroxide solution from the reactor is drawn from the bottom of the lower section by a gear pump. On the inlet side of the pump are provisions for caustic/acid injection by a pH controller, as well as recirculation and fill valves. The gear pump is a Teel Model IV454 with stainless steel housing and viton gears with graphite bushings. It is driven by a DC 1/3 HP variable speed motor with a 5:1 gear reduction in between so as to keep recirculation pressures within 100 psig. We expect to keep the downstream pressures between 35 and 80 psig which correspond to recirculation flow rates of 0.25 to 0.36 gpm respectively. The outlet of the pump is connected to a domestic 5 micron water filter to knock out any re-circulating particulates that would clog the spray nozzle. The clean solution then passes through a valve

combination which permits liquid sampling (to monitor peroxide concentration) during a test and includes a bypass outlet allowing some of the clean solution to pass through a cooling section to provide for temperature control. The cooled liquid is then pumped back to the test chamber facilitating thorough mixing of the solution. The re-circulating fluid then passes through a plumbing tree that houses a pressure gauge and a pH sensor (to provide for pH control), as well as a type T (Copper-Constantan) thermocouple to measure the fluid temperature. The solution is then re-circulated into the top of the test section through an injection nozzle. As a drying cycle progresses, it is expected that water in the exhaust gas from the wood drying kiln will be knocked out in the test cell due to a decrease in gas temperature. In order to accommodate the water being condensed, as well as the caustic being injected into the test cell, an overflow tap at 9" (operating level) from the bottom of the treatment cell has been provided. A dual set of VOC analyzers, to measure the actual VOC reduction between the inlet and the outlet of the cell, allows for measurement of treatment efficiencies. The redesigned chamber is expected to allow for a greater efficiency in VOC reduction and will be safer, less expensive, and easier to apply to an actual sawmill setting.

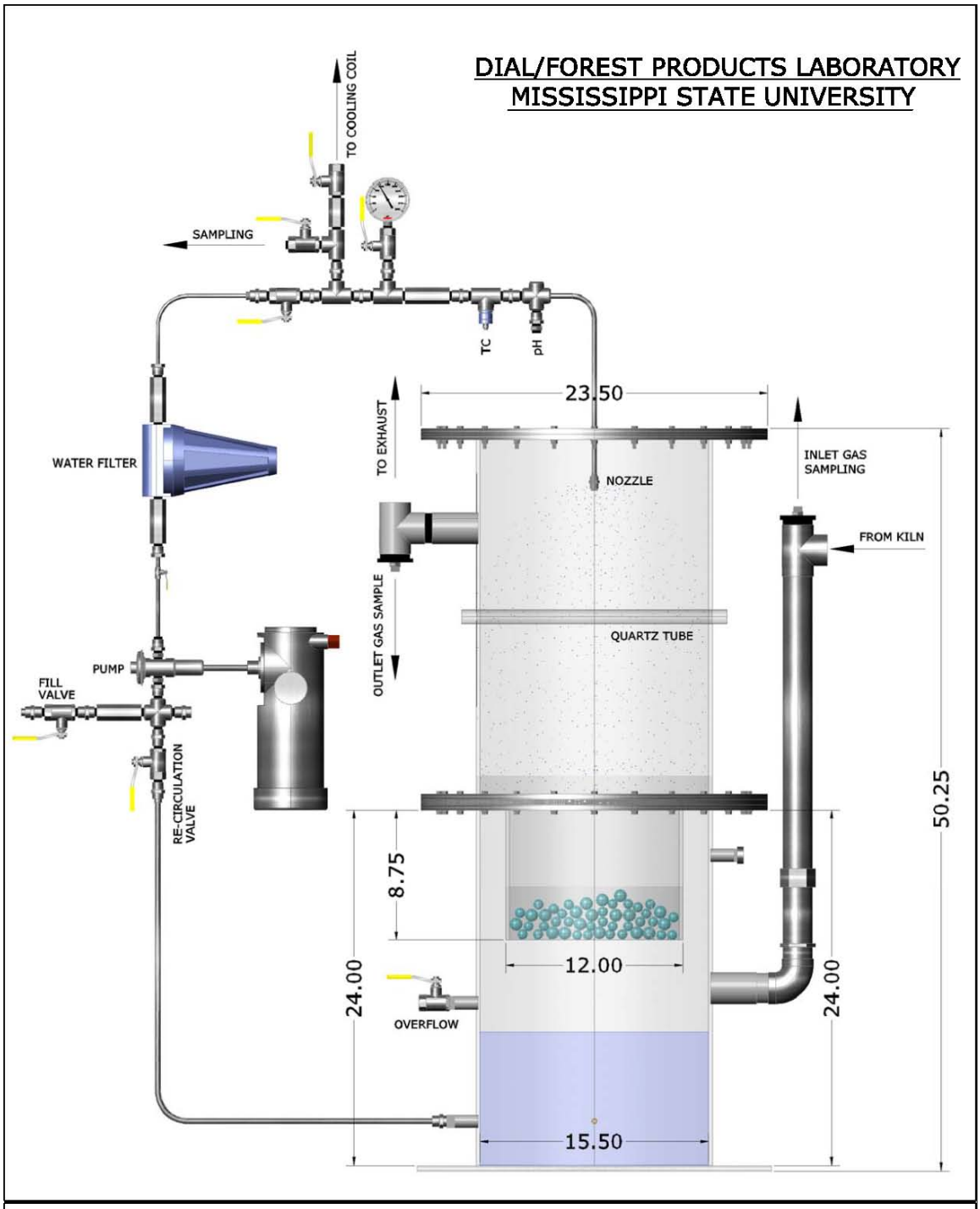

FIGURE 1
VOC TREATMENT FLOW PROCESS

In order to increase the volumetric energy density a secondary treatment chamber was constructed as shown in Figure 2. This secondary chamber was installed downstream of the primary chamber. It consists of a 3 in. diameter, 16 in. long aluminum pipe with a 1 inch diameter quartz tube running at an angle down the middle of it for housing the UV lamp.

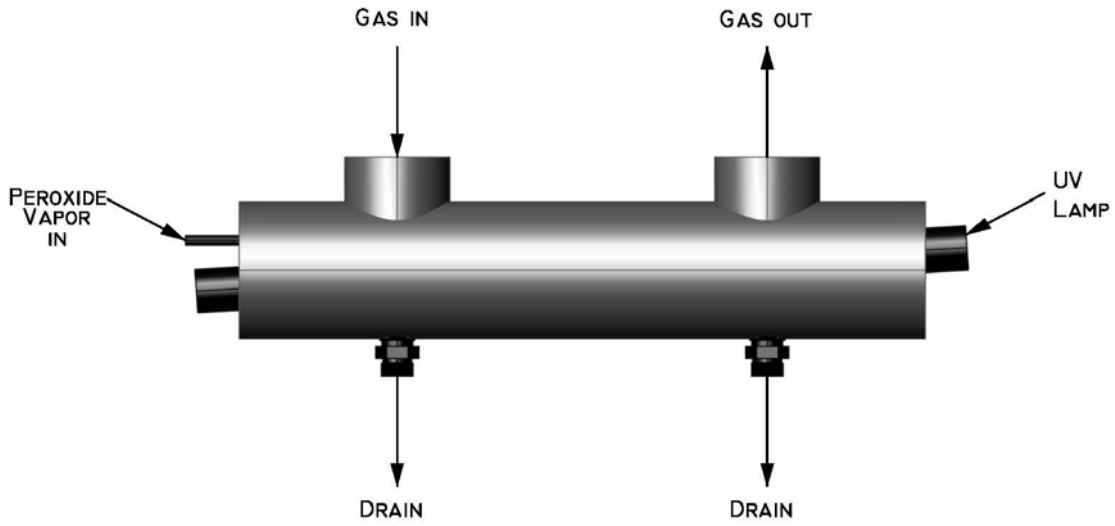

Fig 2: Schematic of secondary treatment chamber

Work Performed

One set of runs was conducted in this quarter. Off-gas from the kiln was routed through the primary treatment chamber to reduce the moisture content as well as quench the gas. The off-gas was then passed through the secondary treatment chamber for VOC reduction. Total hydrocarbon concentrations were measured at the inlet and the outlet of the secondary chamber to determine the VOC destruction efficiency. Hydrogen peroxide was pumped through a stainless steel tube immersed in an oil bath to vaporize the hydrogen peroxide. The hydrogen peroxide vapor was then injected into the secondary chamber to treat the VOC's in the presence of a UV vapor lamp. Hydrogen peroxide concentrations of 3%, 5% and 10% were studied. A low pressure and a medium pressure UV lamp were utilized to enhance the VOC oxidation. A 3% concentration of hydrogen

peroxide vapor dropped the VOC concentration from 864 ppm to 769 ppm, a 11 percent decrease (with the low pressure UV lamp).. The 5% peroxide injection had a similar decrease and turning off the UV lamp brought the downstream concentration up to 810 ppm, a 6.25% decrease. The 10% peroxide solution also had about a 10 % reduction with the low pressure UV lamp in place. With the medium pressure lamp in place, VOC destruction efficiencies were in the 14% range with the upstream concentration of 849 ppm being reduced to 730 ppm.

We also made a presentation at the ITP Forest Products Peer Review in the first week of April in Atlanta. We are operating under a no-cost extension for the current year.

Patents: None at this stage.

Milestone Status Table: (for the first year)

ID Number	Task / Milestone Description	Planned Completion	Actual Completion	Comments
1	**Infrastructure Rebuild and Acquisition**	12/31/04	4/1/05	
1.1	Kiln Repair	12/31/04	12/31/04	
1.2	Peroxide Delivery System	12/31/04		Peroxide addition will be performed for the testing as needed. Titration will be performed during a test to determine the amount of peroxide to be added
1.3	Preliminary Cell Construction	12/15/04	4/1/05	
2	**Process Reaction Conditions**[1]	3/18/05		Delayed.
2.1	Solution pH Sequence	2/18/05		Delayed
2.2	Peroxide Concentration Sequence	2/28/05		Delayed
2.3	UV Light intensity (number of lamps)	3/18/05	7/28/05	Delayed awaiting success in measurements.
3	**Reaction Kinetic Conditions**[1]	7/30/05		
3.1	Droplet Size and Delivery Testing	4/15/05		Delayed
3.2	UV light orientation testing	5/30/05		No longer applies due to new design
3.3	Height of chamber	7/30/05	8/10/05	Smaller secondary chamber completed
4	**Preliminary Process Economics**	8/31/05		Delayed awaiting success in measurements
4.1	Mass Balance	8/20/05		
4.2	Energy Balance	8/20/05		
4.3	Prototype Equipment Requirements	8/20/05		
4.4	Consumable and Labor Estimates	8/31/05		
5	**Prototype design**	9/30/05		Delayed awaiting success in measurements

[1] Test Sequences may be pursued simultaneously depending on preliminary results.

Budget Data (as of date): The actual spending should reflect the money actually spent on the project in the corresponding periods.

The Project Spending and Estimate of Future Spending table shown below reflects the original budget. Revisions to the scope of work and the estimate of Future Spending will be made as the financial situation becomes clearer.

Project Spending and Estimate of Future Spending							
Quarter	**From**	**To**	**Estimated Federal Share of Outlays***	**Actual Federal Share of Outlays**	**Estimated Recipient Share of Outlays***	**Actual Recipient Share of Outlays**	**Cumulative**
			Note 1		Note 1		
3Q04	Start	9/30/04	Note 2		Note 2		
4Q04	10/1/04	12/31/04	49,000	0	4,000	0	53,000
1Q05	1/1/05	3/31/05	68,000	73,347	44,000	6050	112,000
2Q05	4/1/05	6/30/05	68,000	30,911	39,000	147	107,000
3Q05	7/31/05	9/30/05	59,266	26,822	18,932	0	78,198
4Q05	10/1/05	12/31/05	150,000	66,214	75,000	0	225,000
1Q06	1/1/06	3/31/06	175,000	153	65,000	0	240,000
2Q06	4/1/06	6/30/06	175,000	1,112	60,000	0	235,000
3Q06	7/31/06	9/30/06	100,000		60,000		160,000
4Q06	10/1/06	12/31/06					
Etc.							
Totals			**844,266**		**365,932**		**1,210,198**

* Update quarterly

General Note: DOE Laboratory partner spending should not be included in the above table. If a DOE Laboratory is a partner, report their spending and spend plan information in the table below (use separate tables if multiple DOE Laboratories are involved):
Note 1: Leave blank. Only the actual DOE/Cost Share amounts spent through 6/30/04 are needed.
Note 2: Amount for this quarter and subsequent quarters should be updated as necessary on a quarterly basis. Estimates need to be provided for the entire project. If spending for a given quarter is different than estimated, then the remaining quarter's estimates should be updated to account for the difference. Total DOE and Cost Share amounts should be the same as the Award amount.
Note 3: This should match the amount on the SF269A section 10.c. Column III (10.j. Column III on the SF269).
Note 4: This should match the amount on the SF269A section 10.c. Column II (10.j. Column II on the SF269).
Note 5: This should match the amount on the SF269A section 10.b. Column III (10.i. Column III on the SF269).

Note 6: This should match the amount on the SF269A section 10.b. Column II (10.i. Column II on the SF269).
Note 7: This should match the amount on the SF269A section 10.a. Column III (10.d. Column III on the SF269).
Note 8: This should match the amount on the SF269A section 10.a. Column II (10.d. Column II on the SF269).

Low VOC Drying of Lumber and Wood Panel Products

Banerjee: IPST at Georgia Tech

ID13439

PROGRESS REPORT

Project Title: Implementing Strategies for Drying and Pressing Wood Without Emissions Controls

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Other Partners: Lawrence Otwell (Georgia-Pacific)

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Project Objective: The objective of this project is to devise strategies for sizing down control devices needed for treating VOC emissions from dryers and presses for wood products such as OSB and veneer.

Background: Our previous work has brought us to the point where we can dry wood full-scale for brief periods without emissions controls. We now need to do this consistently and without adversely affecting throughput and production economics. Our first objective is to identify the mechanisms of release of some of these HAPs, to identify second-order variables that affect HAPs generation, and to develop and field-demonstrate a comprehensive strategy. Our second goal is to reduce resin use during pressing through droplet control. An ancillary goal is to determine the feasibility of applying urea as a fine mist to the mat in order to quench formaldehyde emissions.

Status:

- The fines content of green furnish was determined.
- The energy savings realized through higher moisture drying was calculated.
- The mechanism behind the relationship between fines generation and knife angle/temperature was studied through pilot and full-scale studies on chipping.
- A new chemical augments the ability of urea to decrease formaldehyde emissions.

Fractionation of Fordyce Flakes

We have previously shown that fines lead to a disproportionate amount of HAPs and it is important to measure the quantity of fines that enter the dryer. It is difficult to measure green fines because of their tendency to stick to green flakes, and measurement of green fines through screening probably leads to an undercount. The fines content of green furnish was measured as follows.

Green flakes received from G-P, Fordyce, were uniformly mixed and separated into 17 bags. Nine of these bags were dried at 50^{o}C to about 95% solids. The others were stored at 4^{o}C. The wet and dried flakes were then fractioned with a Gilson screen at LP's Athens mill. The results are shown in Table 1. The fines average 5.8 and 7.2% for the dry and green flakes, respectively. A t-test showed that the fines populations of the green and dry flakes were different at the 80% confidence level. Each flake (from a total of about one hundred) in the 1 1/2" fraction was then washed in a 5-gallon bucket of water. The washed flakes were then dried at 105^{o}C to obtain the dry weight. The water was screened through a 1/8" screen (to remove large pieces) and then through filter paper. The material trapped on the filter paper was dried and weighed. The results, shown in Table 2, demonstrate that the green flakes have more fines associated with them. Earlier results (October 2003) from LP Athens showed that more fines were associated with green flakes than with dry. The present results suggest that the difference is even higher because more fines stick to green flakes than to dry ones.

Table 1: Fractionation of flakes (g).

percent solids	1 1/2"	3/4"	1/2"	3/16"	1/8"	pan	total	percent fines
dry flakes								
94.8	70	60	40	35	25	15	245	6.1
94.6	135	60	40	40	20	10	305	3.3
95.3	130	80	35	45	25	30	345	8.7
95.3	130	75	45	45	25	15	335	4.5
95.2	110	60	30	40	25	25	290	8.6
95.1	90	110	60	70	35	30	395	7.6
94.9	65	85	45	45	25	10	275	3.6
94.0	85	50	35	40	20	10	240	4.2
94.8	90	90	35	40	20	15	290	5.2
green flakes								
47.9	360	70	30	40	35	40	575	7.0
49.8	270	75	55	55	40	40	535	7.5
47.1	185	145	70	75	50	40	565	7.1
53.2	190	85	60	70	50	50	505	9.9
48.5	300	115	45	60	45	50	615	8.1
39.2	230	50	50	55	45	45	475	9.5
47.1	235	85	45	50	30	20	465	4.3
52.7	205	125	50	70	40	20	510	3.9

Table 2: Fines content (%, od) of large flakes.		
run	dry flakes	green flakes
1	0.30	0.82
2	0.39	0.76
3	0.50	0.69
average	**0.40**	**0.76**

Energy savings from higher moisture drying

Most mills typically dry their furnish to moisture contents of 2% or less. It takes progressively more energy to remove these low levels of moisture. A calculation for spruce using published heat of sorption data (C. Skaar, Water in Wood, Syracuse University Press. 1972) shows that drying from 6% moisture to dryness requires 1,360 BTU/lb of water removed. Drying from 2% to dryness requires 1,433 BTU/lb of water.

Mechanism of fines generation

We previously showed that matching knife sharpness angle to temperature the level of fines generated and thereby decreases HAPs evolution. The technique has been implemented at the Norbord, Guntown mill and at several Georgia-Pacific facilities. We extended the approach to chipping wood for pulp manufacture and determined the mechanism underlying the process. It turns out that the separation of a flake from a log requires both cutting and splitting actions, and these have different temperature dependencies which result in differing proportions of flakes and fines at different temperatures. The splitting action (caused by the knife creating forces perpendicular-to-the-grain) is temperature-dependent but the cutting action is not.

The results have been submitted to *Tappi Journal* for publication. A copy is attached below.

Capture of HAPs from press emissions

In the last quarter a new chemical that traps organics was screened for its ability to reduce press emissions. Several press runs were made and the variability of wood is making it difficult for the pinene reduction to be quantified. However, it is clear that the chemical augments the ability of urea to decrease formaldehyde emissions. Details will be provided in the next quarterly report.

We anticipate that we will need to do about four months of lab work to define cost benefits, after which we will approach Georgia-Pacific for pilot and full-scale work.

Submitted to Tappi Journal

Temperature Effects on Fines Generation during Wood Chipping and Flaking

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Summary

The amount of fines produced from flaking wood for oriented strand board depends on both temperature and the knife sharpness angle. In winter a higher knife angle reduces the fines significantly. Pilot and full-scale work on chipping wood for pulping was conducted in an attempt to determine whether a similar effect applied to pulpwood. An inverse relationship was found between the production of fines and overthick chips at different temperatures, implicating a temperature-dependent property of wood. The separation of a flake from a log requires both cutting and splitting actions, and these have different temperature dependencies which result in differing proportions of flakes and fines at different temperatures. It was shown through literature results and work with high-lignin pulp fibers that the splitting action (caused by the knife creating forces perpendicular-to-the-grain) is temperature-dependent but that the cutting action is not.

Application Statement

Matching knife sharpness angle to temperature may lead to wood savings.

Introduction

Flaking wood for the manufacture of oriented strand board (OSB) creates a substantial proportion of fines which is detrimental from both a wood yield and an emissions perspective. Fines release a disproportionate amount of formaldehyde, methanol, and other "Hazardous Air Pollutants" during flake drying (1). They also consume large amounts of resin. We have previously demonstrated that fines generation during commercial flaking depends, in part, on a combination of knife angle and wood temperature (2). Figure 1 demonstrates that use of a larger knife angle during cooler temperatures reduces the amount of fines produced, a practice that has been implemented at several mills. A temperature of 20°C appears to be the cross-over point for changing from one knife angle to another. In this paper we explore the mechanism underlying this phenomenon and extend it to chipping wood for pulping.

Methods and Materials

Experiments were conducted in two parts. In the first part of the study we examined the chipping of southern pine logs to see if the temperature sensitivity seen for OSB flakes would also appear in the production of thicker pieces of wood. We followed this study with a measurement of handsheet strength properties to see whether the temperature sensitivity observed might be attributable to wood constituents.

Pilot-scale chipping was done with fresh Southern yellow pine logs (60 x 12-cm diameter) that were hand-debarked and soaked in water at three temperatures (6, 21 and 42°C). The logs were chipped using a Carthage disk chipper at four knife angles (32, 34, 36 and 38 degrees) and the chips were screened. Each experiment with a given combination of knife angle and wood temperature was replicated four times. Additionally, full-scale chipping data were collected from a mill in Georgia which uses a 296-cm Carthage chipper with a 33.5° knife angle. These data were examined to see if there were indications of relationships between temperature and chip fractions. The results reported are based on averages of almost a thousand determinations made during each quarter in 2004.

Wet tensile measurements were made from southern pine kraft pulps with various kappa numbers. Each pulp was beaten in a PFI mill to several freeness levels and disintegrated in a British disintegrator, and eighteen standard TAPPI handsheets were prepared from each pulp Two strips (2.5 cm wide) were cut from each handsheet and rewetted in pans maintained overnight in temperature-controlled rooms set at 4, 10, 23, and 32°C. The strips were soaked for one hour and the excess water was blotted out. After conditioning the handsheets overnight in the temperature-controlled rooms, wet zero-span tensile strengths were measured at three different temperatures.

Results and Discussion

Chipping

The pilot-scale results are presented in Table 1 and the chip size distributions are illustrated in Figure 2. Fines were affected by temperature to a lesser degree for the chips than had been observed for OSB flakes (refer to Figure 1); instead of the approximately 3–7% differences seen in the flaker fines, chipper pins and fines varied only by 2.1 to 3.8% with temperature for any given knife angle. As Figure 2 shows clearly, the width of the chip size distribution does not appear to be dependent on knife angle. Our results for the pins-and-fines and the oversize fractions are illustrated in Figure 3. The population of the 45 mm chips is higher than those found in a commercial chipper, which would include a card breaker and would also use thicker logs. The proportion of the fines in the pins-and-fines is small and a plot of the pins alone shows the same trends. Changes in the knife sharpness angle (at constant temperature) appear to cause (at most) about 3.5% differences in the amounts of pins and fines. As Smith and Javid (3) point out, fines (<3 mm) and >45 mm fractions are influenced more by wood characteristics than by chipper performance, and we likewise noted that there was significantly lesser variation in this fraction than in the pins (>3 mm) fraction. We found that changes in knife angle caused slight differences (ranging from 0 to about 3% maximum) in the amount of overthicks (>45 mm) produced.

Stuart and Leary (4) examined factors affecting chip size distribution of chips produced at sawmills, and they concluded that knife angle was of lesser significance to chip fractions than

variables such as temperature and disk speed. In their study they defined overthicks as chips over 8 mm thick. Figure 3 suggests that the generation of fines and oversize chips is inversely related. Stuart and Leary (4) found some evidence of this but also noted that the evidence was inconclusive. For further examination of this question, we collected data for one year from a mill in Georgia (in the southeastern U.S.) that processes both hardwood and softwood. The inverse relationship between fines and oversize material in this mill (as shown in Figure 4) is compelling. Clearly, there are fewer fines and more oversize material in the third quarter when the wood temperature reaches a maximum.

Wallace et al. (5) reported an inverse relationship between pin chip production and temperature between -1 and 27°C, but the relationship between oversize chips and increasing temperature was not explored. They calculated that pin chip generation would increase by 0.1–0.2% for every 1°F drop in ambient temperature. Our Figure 4 results are roughly consistent with this estimate, though the trends may not be linear over the entire temperature range examined in our experiments. Hernandez and Quirion (6) and Hernandez and Lessard (7) have also found that fewer oversize chips and more pins and fines are produced in the winter when the wood was frozen. They concluded that the temperature sensitivity of the mechanical properties of wood (the modulus of rupture especially) lead to a difference in the ratio of parallel-to-the-grain strength to the perpendicular-to-the-grain strength. They reasoned that this ratio would be higher in the winter than in the summer, resulting in thinner chips in cold conditions. Relationships between pins and oversize chips have also been induced by parameters other than temperature; like Stuart and Leary, Smith and Javid (3) also reported that higher chipper speeds increased pins and fines.

The inverse relationship between fines/pins and oversize chips in Figure 4 demonstrates that the fines are not created by random frictional forces but by a temperature-related parameter such as the flexibility of wood. Friction (perhaps influenced by moisture content) may be a contributor, but it is not dominant. Our conclusion from the chipping work is that fines generation depends on a temperature-dependent property.

The process of separating a chip or a flake from a log must involve some combination of cutting (forcing a knife parallel to the grain) and splitting (tearing the wood apart perpendicular to the grain ahead of the knife tip). In a laboratory simulation of chipping, Uhmeier (8) found that the forces parallel and perpendicular to the blade can vary considerably depending upon the clearance angle (among other factors). Under some conditions the two forces are roughly equal. Changing the knife angle must change the proportion of cutting and splitting involved; a larger angle would increase the splitting component. The cutting and splitting actions are generated by the parallel and perpendicular forces acting on the wood at the knife interface; this means that changing the knife angle will also alter the proportion of the fines generated. If the cutting and splitting forces have different temperature dependencies, and if knife angles have different ratios of cutting *vs.* splitting action for each temperature, then every knife angle should have a different cause-and-effect relationship (slope) with temperature for fines. This is indeed what is observed in the experimental data (see Figure 1).

Handsheet Testing

Splitting is likely to occur through fractures in the middle lamella region where there is a high lignin content. Lignin softens with increasing temperature and the force required for split-

ting should, therefore, drop proportionately. The cutting action is more difficult to evaluate. To some degree cutting involves the rupture of fiber, and an estimate of how the cutting forces are influenced by temperature would require measurement of the mechanical properties of wood fibers parallel to the grain at different temperatures. The temperature ranges in our experiments (Figure 1) are not large enough that consideration would usually be given to differences in mechanical properties, and the intrinsic variability of wood would likely make it difficult to observe any differences in chip fraction distributions due to corresponding changes in mechanical properties parallel to the grain. For a more controlled experiment, we chose to use pulp fibers to simulate wood fiber (more homogeneous) and measured the wet tensile strength of handsheets from that fiber as a function of temperature. High- and low-kappa pulps (*i.e*., high- and low-lignin pulps) were made into handsheets to determine whether tensile properties are temperature- sensitive, and if so, whether more temperature-sensitivity comes about from papers with higher proportions of lignin or cellulose. Because fiber chemistry will affect bonding we expected that the absolute tensile values would be different, but we made the assumption that the presence or absence of strength trends with temperature would be indicative of material behavior by the lignin or cellulose in solid wood. High-lignin pulps were also expected to contain higher percentages of the original hemicellulose content than the low-lignin pulps.

In a zero-span measurement a handsheet is pulled apart with no space allowed between the jaws of the clamp (9). The result measures the strength of a collection of individual fibers and we considered this force to be analogous to the cutting action in wood. Values for the zero-span tensile index are reported for pulps beaten to different freeness levels in Table 2. The 100-kappa pulp has the highest lignin content. No overall trends are apparent; the zero-span tensile index is independent of temperature across the temperature range. In contrast, the finite-span tensile index (for the rupture of fiber networks) decreases with increasing temperature (10). If our analogy between pulp and wood fiber is valid, then we would conclude that the cutting action of wood is also temperature-independent over the temperature range considered. Therefore, splitting must be the temperature-sensitive component of the chipping process. This difference accounts for the trends seen in Figure 1. Furthermore, the results are consistent with and supportive of the work of Hernandez and coworkers (6, 7).

Conclusions

While both knife angle and wood temperature influence fines generated from stranding wood for OSB manufacture, the knife angle is of lesser importance in chipping logs for pulping. Full-scale data show that the production of pins/fines and oversize chips are inversely related through the seasons, most likely because of temperature variations. Zero-span with handsheets indicate that fiber strength (related to the cutting action parallel to the grain of wood) is temperature-independent.

The results suggest that fines from wood chipping and flaking depend upon both temperature and knife angle, although the effect of knife angle on chipping is very difficult to demonstrate in the absence of extensive data. Nevertheless, given that knife changes are of essentially no cost and that the knife angle effect clearly exists for flaking and is supported by theory, it should be well worth making such a change for chipping. This change will be most effective in colder climates and could be made as infrequently as twice per year when the temperature crosses the 20^{o}C threshold.

Acknowledgment

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References

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Table 1: Chip distribution (%) at various knife angles and temperatures[1].

	32° knife angle			34° knife angle			36° knife angle			38° knife angle		
screen (mm)	temperature (°C)			temperature (°C)			temperature (°C)			temperature (°C)		
	42.2	20.6	5.6	42.2	20.6	5.6	42.2	20.6	5.6	42.2	20.6	5.6
< 3 (fines)	0.8 (0.3)	0.8 (0.3)	1.0 (0.2)	0.3 (0.1)	0.5 (0.2)	0.8 (0.4)	0.3 (0.1)	0.3 (0.1)	0.7 (0.4)	0.3 (0.1)	1.0 (0.5)	0.8 (0.2)
> 3 (pins)	2.4 (1.0)	2.7 (1.3)	6.0 (1.2)	2.8 (1.2)	3.1 (1.3)	4.7 (1.7)	2.0 (1.0)	1.7 (0.5)	3.7 (1.6)	2.2 (0.8)	4.3 (0.7)	5.4 (1.5)
> 7	68 (11)	65 (10)	84 (2)	64 (14)	69 (9)	75 (4)	63 (12)	54 (12)	69 (5)	58 (11)	71 (5)	72 (3)
> 8	25 (10)	28 (10)	9.4 (1)	28 (11)	25 (8)	18 (6)	32 (11)	40 (9)	26 (7)	37 (9)	23 (6)	22 (4)
> 45	4.1 (2.6)	3.1 (2.4)	0.2 (0.2)	5.8 (4.8)	2.6 (1.7)	0.9 (0.7)	2.7 (2.4)	4.4 (2.8)	0.8 (0.3)	2.7 (1.9)	1.0 (0.5)	0.3 (0.3)

[1]average of four determinations, average deviation in parentheses.

Table 2: Effect of temperature on zero-span tensile index[1].					
kappa no	CSF (ml)	**4 ^{0}C**	**10 ^{0}C**	**23 ^{0}C**	**32 ^{0}C**
85	430	108 (2)	118 (4)	112 (3)	108 (5)
85	370	136 (6)	149 (5)	138 (3)	107 (5)
100	463	130 (4)	121 (5)	117 (4)	118 (4)
100	370	105 (2)	118 (5)	109 (3)	107 (5)
100	202	96 (4)	117 (6)	92 (2)	105 (4)
100	152	93 (3)	106 (5)	93 (6)	115 (4)
[1]in Nm/g; the values in parentheses are standard deviations					

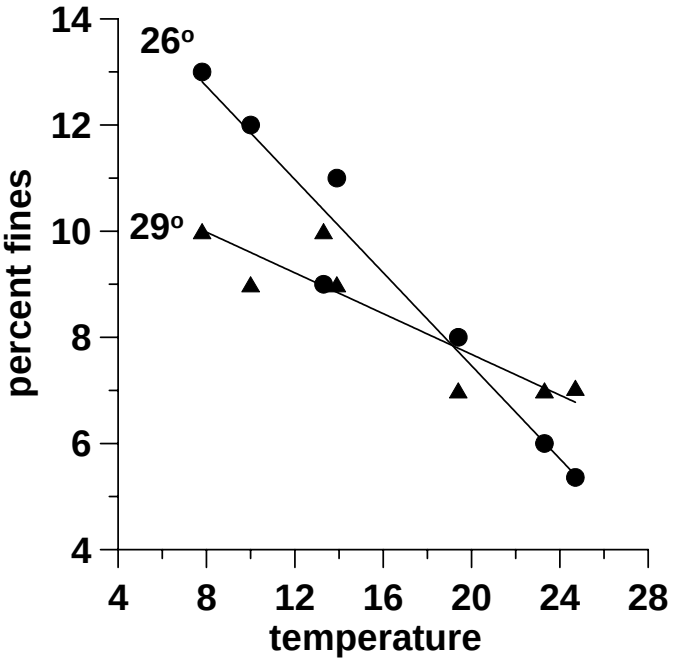

Figure 1: Dependence on fines generation on ambient temperature during commercial flaking.

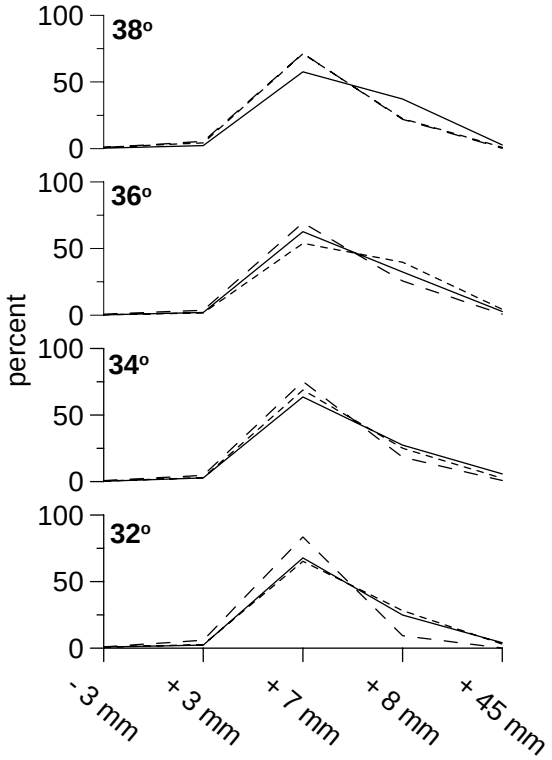

Figure 2: Distribution of chips generated during pilot chipping. Each line is at a different temperature (42, 21 and 6°C).

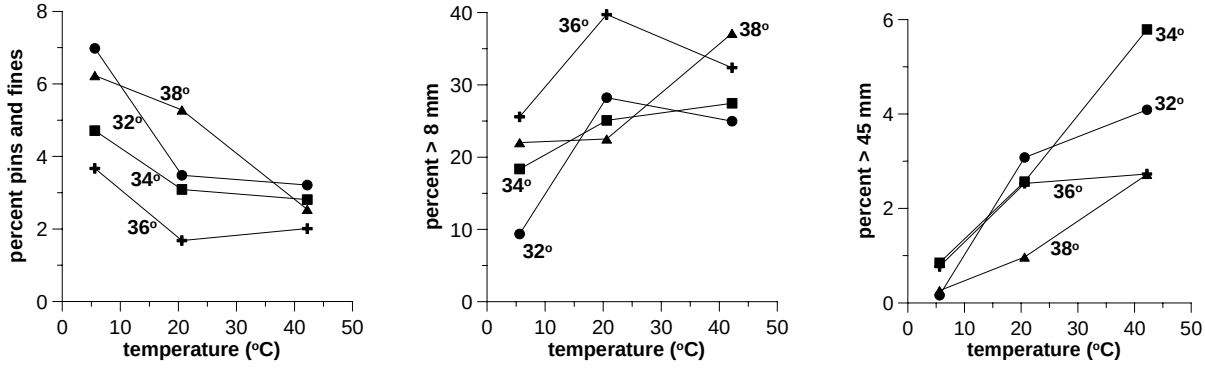

Figure 3: Dependence of the generation of pins-and-fines (left) >8-mm (center) and >8-mm (right) chips on log temperature.

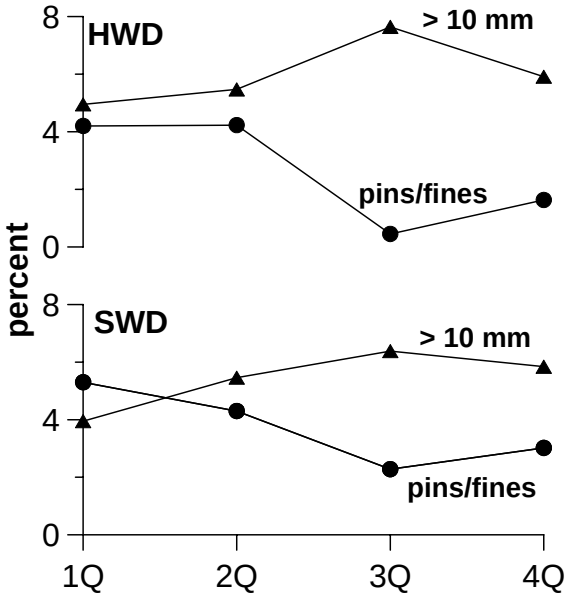

Figure 4: Pins & fines and oversize chips generated at a Georgia mill. The average temperatures for 1Q-4Q were 10.9, 23.3, 26.6 and 15.1^{o}C, respectively.

ID Number	Task/Milestone Description	Planned Completion	Percent Completion	Actual Completion	Comments
1	Identify factors that lead to HAPs generation during drying				
1.1	Lab work	4.04	100	6.04	
1.2	OSB mill trials (rotary dryers)	5.05	100	3.04	
1.3	Veneer mill trials	12.04	100	6.03	
1.4	OSB mill trials (conveyor dryers)	5.05	100	12.04	
2	Integrate items identified above into strategies for reducing emissions during drying				
2.1	Lab work				1
	(a) Construct lab-scale flaker for high-speed imaging of fines production	6.05	100	12.05	
	(b) Devise lab tests for generating input parameters for model	12.05	100	3.06	
	(c) Model fines production from flaker	3.06	100	6.06	
	(d) Pilot trials at LP, Nashville	9.05	100	6.05	
2.2	OSB mill trials				
	Skippers (rotary dryer)	6.06	20		2
	Dudley (rotary dryer)	3.06	10		2
	Guntown (conveyor dryer)	12.05	100		3
	Fordyce (rotary dryer)	9.05	100		
2.3	Veneer mill trials	12.04	100	6.03	
2.4	Work with flaker vendor for broad commercialization	3.07	0		
3	Extend above strategies to pressing OSB and veneer	10.04	100		4
4	Reduce press HAPs by applying urea and resin as a fine aerosol				5
4.1	Lab work with urea/resin	12.05	30		
4.2	Effect on product properties	6.06	0		
4.3	Pilots	12.06	0		
5	Studies on knife corrosion				
5.1	Rate studies and characterization	3.05	100	12.04	
5.2	Application of coatings	6.05	100	12.04	
5.3	Field trials	9.05			6
6	Economic analysis	12.06	0		
7	Final report	3.07			

Comments

1. Flaking was found to be the principal cause of fines that lead to HAPs upon drying and this element of the project was developed further. An understanding of the process is needed so that the results from the field (which are presently successful) can be applied with confidence.
2. Promulgation of the wood MACT rule was delayed by EPA and the mill trials were pushed back by Georgia-Pacific.
3. Sampling must be done across at least four seasons so that the effect of wood temperature can be modeled.
4. Work on pressing was terminated, and the resources applied to drying where a bigger return is anticipated. This was done upon the advice of our industry advisory board.
5. Work on this item was delayed so that the dryer aspect of the work could be accelerated as discussed above.
6. The Norbord mill does not want to run a field trial because of safety reasons.

Project Spending and Estimate of Future Spending DE-FCO7-96ID13439							
Quarter	**From**	**To**	**Estimated Federal Share of Outlays***	**Actual Federal Share of Outlays**	**Estimated Recipient Share of Outlays***	**Actual Recipient Share of Outlays**	**Cumulative**
	Start	9/30/04		1,595,592		651,059	
4Q05	10/1/05	12/31/05	50,000	23,195			
1Q06	1/1/06	3/31/06	90,000	40,519			
2Q06	4/1/06	6/30/06	40,000	47,219			
3Q06	7/1/06	9/30/06	90,000				
4Q06	10/1/06	12/31/06	40,000				
1Q07	1/1/07	3/31/07	35,715				
Totals			**605,715**	**1,706,525**		651,059	
Approved (committed)			**1,998,007 (1,792,383)**				

VOC and HAP Recovery Using Ionic Liquids

Milota: Oregon State University

ID14432

QUARTERLY PROGRESS REPORT

Project Title: VOC and HAP Recovery Using Ionic Liquids

Covering Period: April 1 – June 30, 2006

Date of Report: July 18, 2006

Recipient:
Oregon State University
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119 Richardson Hall
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Award Number: DE-FC07-03ID14432

Subcontractors: None

Other Partners: Weyerhaeuser, Inc., Federal Way, WA; Louisiana Pacific Corp., Portland, OR; Boise Cascade Corp., Boise ID.

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Project Objective: Synthesize RTILs and optimize their chemical structures for a VOC/HAP absorption process. Evaluate of the solubility and removal of VOC compounds in the synthesized RTILs and determine other chemical and physical properties important for absorption. Construct a continuous prototype absorption system using the RTIL most suited for VOC removal. Using the prototype, determine the ability of the RTIL to clean exhaust during long-term trials on wood dryer and press exhaust.

Background: During the manufacture of wood composites, paper, and to a lesser extent, lumber, large amounts of volatile organic compounds (VOCs) such as terpenes, formaldehyde, and methanol are emitted to air. Some compounds are also hazardous air pollutants (HAPs). The air pollutants produced in the forest products industry are difficult to manage because the concentrations are very low. Presently, thermal oxidizers (TOs) are commonly used for the destruction of VOCs and HAPs. TOs consume large amounts of natural gas to heat air and moisture. The combustion of natural gas generates increased CO_2 and NO_x, which have negative implications for global warming and air quality.

Many shortcomings of TOs and other control technology are addressed by an absorption system containing a room-temperature ionic liquid (RTIL) as an absorbent. RTILs are salts, but are in the liquid state at room temperature. RTILs, an emerging technology, are receiving much attention as replacements for organic solvents in industrial processes with significant cost and environmental benefits. Some of these processes include organic synthesis, extraction, and metal deposition. RTILs would be a excellent absorbents for exhausts from wood products facilities because of their unique properties: no measurable vapor pressure, high solubility of wide range of organic compounds, thermal stability to 200°C, and immiscible with water.

Preliminary results at OSU reveal that fatty acids and resins could dissolve in RTILs, although the solubility has yet to be quantified. A phase separation between a fatty acid and a RTIL was seen when a large amount of fatty acids was added to a RTIL.

Status: During the first few weeks of the quarter we completed the lab analyses on the mill trial data. That data was reported in the previous quarterly report. During this quarter we have disassembled the continuous absorption device and began construction on a larger column. This is mostly completed. We have also done a significant amount of method development to be able to measure additional HAPs during the mill trials. In addition to methanol and formaldehyde, we should be able to quantify acrolein, acetaldehyde, and propionaldehyde. We are arranging for mill trial locations and have two sites tentatively identified. We have visited the sites and measured for equipment. The hold up at this point ionic liquid. We opted to purchase the additional ionic liquid and are having difficulties getting the supplier to deliver. Two papers were written and submitted for publication in refereed journals.

Plans for Next Quarter:

During the next quarter we plan to complete the larger columns and make preparations for additional (final) mill studies. If the balance of the ionic liquid is delivered in August,

we can be at the mill before the end of September. We are almost completed with task #10 with tasks #11, #12 and #13 remaining.

Patents: None.

Publications/Presentations:

Milota, M., P. Mosher, and K. Li. 2007. VOC and HAP removal from dryer exhaust gas by absorption into and ionic liquid. Submitted to Forest Products Journal 7-18-06

Wang, Fujun, M.R. Milota, Paul Mosher, Kaichang Li, and Michelle Yankus. 2007. Henry's Law constants for methanol and α-pinene in ionic liquids. Submitted to Wood and Fiber Science 6-14-06.

Milota, Michael R. And Kaichang Li. 2006. VOC and HAP Recovery using Ionic Liquids. Industrial Technologies Program Forest Products Peer Review. Atlanta, GA. April 4-5, 2006 (Presentation and poster).

Wang, Fujun. Henry's Law Constants for Pollutants from the wood industry in room temperature ionic liquids. M.S. Thesis. Oregon State University. August, 5, 2005

Milota, Michael R. and Kaichang Li. 2004. Preliminary Work on VOC and HAP Recovery Using Ionic Liquids. In:2004 Paper summit, Spring Technical and International Environmental Conference. May 3-6. Atlanta, GA (pp. Electronic proceedings).

Milota, Michael R. And Kaichang Li. 2004. Preliminary Work on VOC and HAP Recovery Using Ionic Liquids. Tappi Paper Summit. Atlanta, GA, May 3-5.

Milestone Status Table: This should be a complete list of project milestones, anticipated completion dates and actual completion dates. The milestone identification number should correspond to the task numbers in your agreement to aid in tracking (example below).

ID Number	Task / Milestone Description	Planned Completion	Actual Completion	Comments
1	SPME / GC Calibration	06/30/03	06/31/03	100%
2	Method for making standards	06/31/03	08/30/03	100%
3	Method for testing equilibrium	08/30/03	12/31/03	100%
4	Ionic liquids	03/31/05	6/15/05	100%
5	Equilibrium testing	12/31/03	03/31/04	100%
6	Test ability to clean ionic liquid	03/31/04	06/30/04	100%
7	Construct lab device for continuous testing	3/31/04	9/30/05	100%
8	Test continuous device on lab exhaust	11/30/04	1-31-06	100%
9	Test continuous device on mill exhaust	02/28/04	3-31-06	100%
10	Construct larger absorber	7/31/05		90%
11	Test large absorber on lab exhaust	9/30/05		0%
12	Mill test 1 of large absorber	12/21/05		5%
13	Mill test 2 of large absorber	3/31/06		5%

Budget Data (9/30/04): The actual spending should reflect the money actually spent on the project in the corresponding periods.

Project Spending and Estimate of Future Spending							
Quarter	**From**	**To**	**Estimated Federal Share of Outlays***	**Actual Federal Share of Outlays**	**Estimated Recipient Share of Outlays***	**Actual Recipient Share of Outlays**	**Cumulative**
	Start	9/30/04	Note 1	113,712.02	Note 1	13,303.44	127,016.46
4Q04	10/1/04	12/31/04	30,000	37,759.77	2000	5,283.88	170,060.11
1Q05	1/1/05	3/31/05	21,359	1,035.18	11000	2,431.08	173,526.37
2Q05	4/1/05	6/30/05	20,000	21,019.40	3000	4,246.48	174,343.62
3Q05	7/1/05	9/30/05	30,000	11,651.05	20000	3,445.41	185,994.67
4Q05	10/1/05	12/31/05	33,750	11,422.41	40376	5,437.57	197,417.08
1Q06	1/1/06	3/31/06	20,000	14,424.79	3000	4592.38	211,841.87
2Q06	4/1/06	6/30/06	15000	14,562.57	0	0	226,404.44
3Q06							
4Q06							
1Q07							
Totals			**268,821**	**Note 3**	**92,680**	**Note 5**	**Note 7**

* Update quarterly

General Note: DOE Laboratory partner spending should not be included in the above table. If a DOE Laboratory is a partner, report their spending and spend plan information in the table below (use separate tables if multiple DOE Laboratories are involved):
Note 1: Leave blank. Only the actual DOE/Cost Share amounts spent through 6/30/04 are needed.
Note 2: Amount for this quarter and subsequent quarters should be updated as necessary on a quarterly basis. Estimates need to be provided for the entire project. If spending for a given quarter is different than estimated, then the remaining quarter's estimates should be updated to account for the difference. Total DOE and Cost Share amounts should be the same as the Award amount.
Note 3: This should match the amount on the SF269A section 10.c. Column III (10.j. Column III on the SF269).
Note 4: This should match the amount on the SF269A section 10.c. Column II (10.j. Column II on the SF269).
Note 5: This should match the amount on the SF269A section 10.b. Column III (10.i. Column III on the SF269).
Note 6: This should match the amount on the SF269A section 10.b. Column II (10.i. Column II on the SF269).
Note 7: This should match the amount on the SF269A section 10.a. Column III (10.d. Column III on the SF269).
Note 8: This should match the amount on the SF269A section 10.a. Column II (10.d. Column II on the SF269).

An Innovative Titania-Activated Carbon System for Removal of VOCs and HAPs ... with In-Situ Regeneration Capabilities

Mazyck: University of Florida

ID14437

PROJECT INFORMATION & PLANNING REPORT

Project Title: An Innovative Titania-Activated Carbon System for Removal of VOC's & HAP's from Pulp, Paper, Paperboard Mills, and Wood Products Facilities with In-Situ Regeneration Capabilities

Covering Period May 1, 2006 through July 31, 2006

Date of Report: July 31, 2006

Recipient:
University of Florida
PO Box 116450 Gainesville, FL 32611
5th Congressional District

Award Number: DE-FC36-03ID14437

Subcontractors:
MicroEnergy Systems, Inc.
300 Industrial Drive Oakland, MD 21550
Rick Sheahan
(301) 334-3455
6th Congressional District.

National Council for Air and Stream Improvement
PO Box 141020 Gainesville, FL 32614
Ashok Jain
(352) 377-4708
5th Congressional District

Other Partners: None

Contact(s):
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Angela Lindner, (352) 846-3033, alind@eng.ufl.edu
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Project Team:
Joe Springer (Project Manager), Golden Field Office
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1617 Cole Boulevard, Bldg 17/2
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Fax: 303-275-4753

Project Objective: The goal of the proposed work is to develop a cost-effective and reliable air pollution control system to remove VOCs (volatile organic compounds) and HAPs (hazardous air pollutants) in emissions from pulp, paper and paperboard mills, and solid wood products facilities. The focus of our proposed control system is a novel composite material of activated carbon coated with a photocatalyst titanium dioxide (TiO_2), herein referred to as TiO_2-coated activated carbon.

Background: Forest products provide essential resources for human civilization, including energy and materials. Compared to fossil fuels, such as coal and oil, the resources from forest products are more sustainable and diverse. In processing forest products, however, unwanted by-products, such as VOCs and HAPs, are generated. Effective control of these emissions is of seminal importance to the continuing development of the forest product industry.

Currently thermal oxidation is the most commonly applied technique for the control of VOC and HAP emissions from the forest products industry sources. While effective, these measures require a constant fuel supply to support the thermal energy requirements. Considering its operating cost involving intensive resources and the formation of NOx, thermal oxidation is not favorable in the long run. In certain facilities, the gas stream is directed to a boiler for treatment. Although the fuel cost is reduced in comparison to thermal oxidation technologies, the transport of the gas stream and the ductwork building is still costly. Hence, a cost-effective technique for in-situ treatment of these pollutants is needed.

Status: This quarter's activity focused on developing a second generation pilot-scale reactor to test the silica-titania coated chemical packing material. Laboratory experimentation focused on biologically active activated carbon for methanol removal. In addition, data collection for the life cycle analysis (LCA) continued.

Pilot Studies

A second generation pilot unit was fabricated for the testing of the silica-titania coated packing material (STCP). This second generation pilot unit (3-D models are shown in Figures 1 and 2) was designed so that the UV lamps are positioned in a horizontal configuration. Since the bulbs in the original reactor had a vertical orientation, the air flow could possibly "short circuit" through bed without seeing high intensity UV light (i.e., without coming in close contact with silica-titania pellets close to the UV lamp). The horizontal configuration in the second generation reactor should help further reduce this possible short-circuiting. The spacing of the lamps in the second generation reactor is similar to that of the original pilot reactor. However, an additional improvement incorporated into the second generation reactor is that lamps can be easily removed in order to change the spacing between the UV lamps, which is an important test parameter for the STCP since the goal is to not only increase performance of the reactor but also reduce the number of UV lamps required in a system. A picture of the second generation reactor is shown in Figure 3. A picture of the electrical cabinet, which houses the ballasts that drive the lamps in the reactor, is shown in Figure 4. This reactor will be tested next quarter with the STCP material (which is currently being produced).

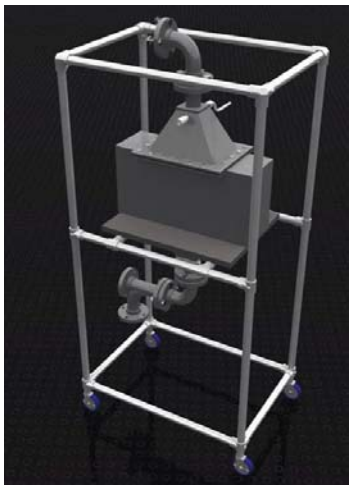

Figure 1. 3-D Model of the Second Generation Pilot Unit

Figure 2. Cut Away View

Figure 3. Photo of the Pilot Unit

Figure 4. Photo of the Electrical Cabinet for the Pilot Unit

Recent work on a biological activated carbon (BAC) system has focused on the conclusion of a 140-day trial to demonstrate feasibility of a lab-scale activated carbon biofilter for removal of methanol in an air stream. Subsequently, assessments of the genetic diversity of bacteria colonizing the biofilter are also in progress.

Feasibility Study for Air-phase Biofiltration of Methanol

The lab-scale air phase BAC reactor was constructed of a 24 inch long by 2 inch diameter clear PVC column containing a mixed packing with Westvaco Bionuchar granular activated carbon

(GAC), perlite, Osmocote slow release ammonium nitrate pellets, and Agrasoke water crystals, in a 4:2:1:1 ratio by volume, on an air-dry basis. The biofilter was inoculated with a well-characterized bacterial culture collected from a Florida paper and paperboard plant. This culture had previously been tested to demonstrate degradation of methanol up to 10,000 mg/L in liquid, rapid growth, and morphological diversity. An uninoculated column, called the control column, was also prepared in the same manner as the biofilter but without bacterial inoculum. Both columns were maintained at ambient temperature and fed methanol at loading rates ranging from 1-18 g/m3 packing/hr in an upflow air stream with 90-95% humidity. Residence time in both columns was 5 minutes during initial high loading and startup, but reduced to 80 seconds during normal operation and lower mass loading rates. A schematic of this experimental system is shown below in Figure 5, and results from this trial are shown in Figure 6.

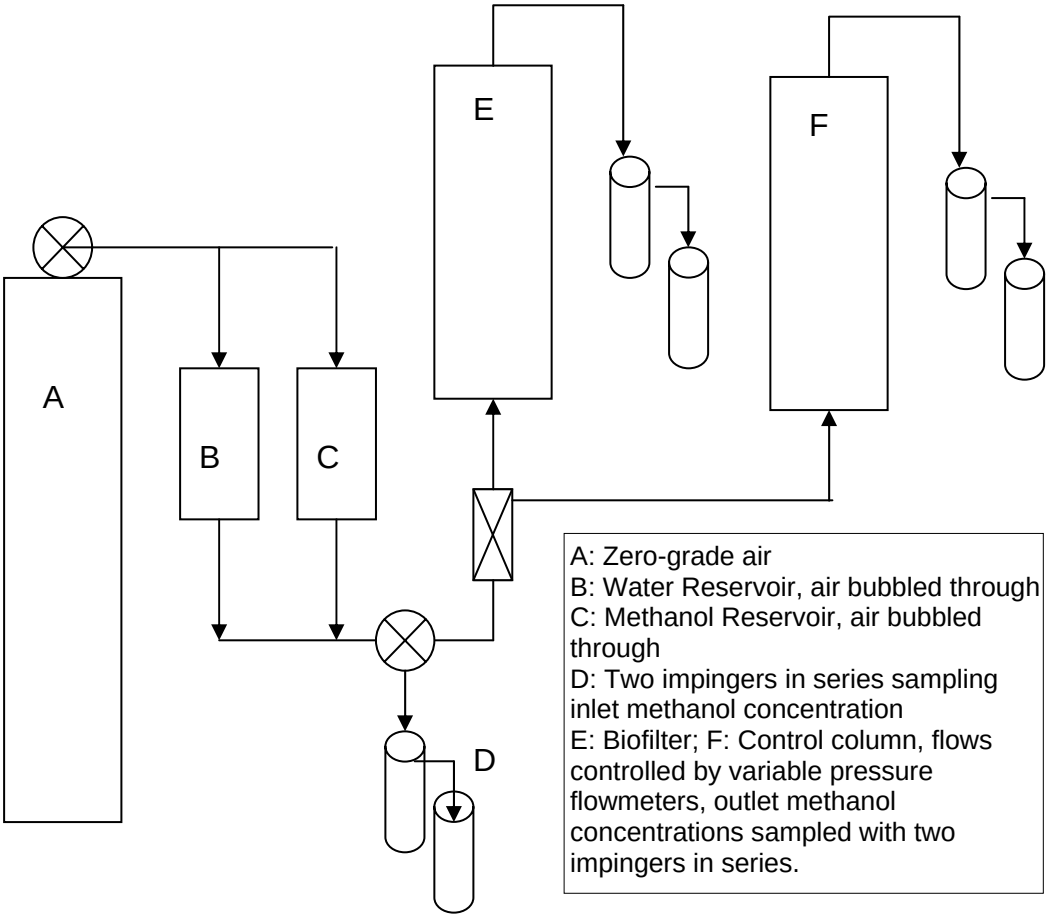

Figure 5. Biofilter Test Stand Set-up

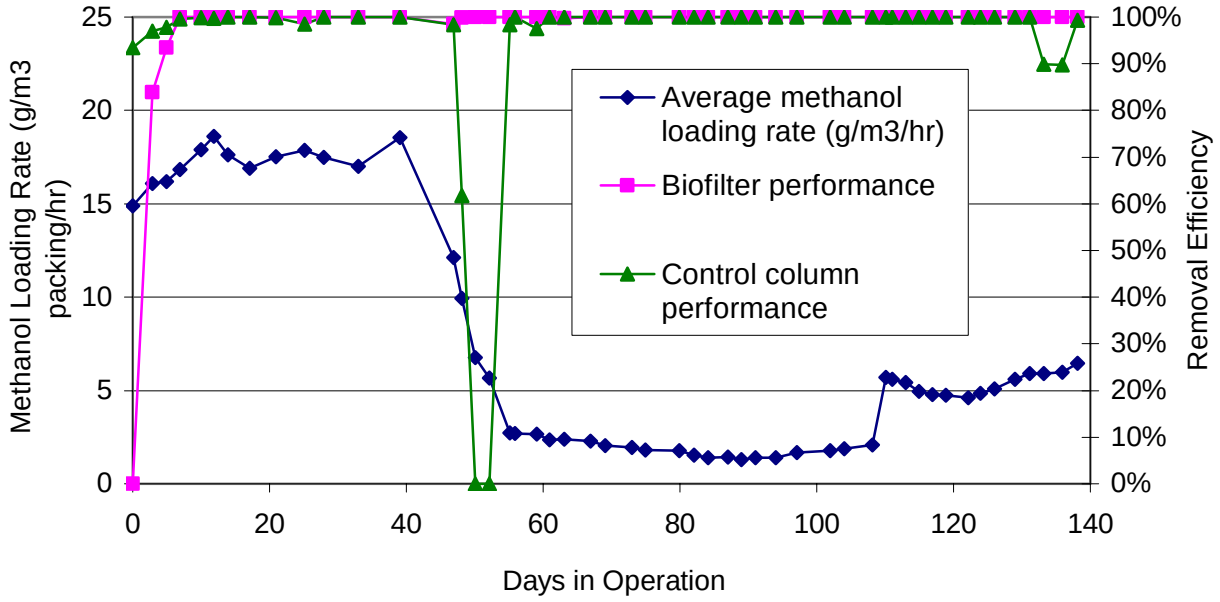

Figure 6. Biofilter Performance

As shown in Figure 6, the biofilter performance reached 100% removal of methanol after 3 days of operation, and remained at or near 100% during the 140 days of operation, during which the loading rates were varied significantly. Initially, the biofilter was operated at very high methanol mass loading rates, about 18 g/m^3 packing/hr and lower residence times, about 5 minutes, during which the adsorptive capacity of the activated carbon could be reached and the bacterial inoculum could be acclimated rapidly to very high methanol concentrations. Despite attempts to maintain sterile conditions during this time, the control column appeared to become contaminated with fungal growth, and also performed at 100% methanol removal, indicating that such a system could be effective without extensive work in cultivating and characterizing a methanol-degrading bacterial consortium.

After this initial high concentration acclimation period, the loading rate was reduced to about 1 g/m^3 packing/hr and a residence time of 80 seconds, more typical of what might be expected in an industrial setting. At this point, the control column went through a period of about 3 days in which little or no methanol was removed, perhaps due to desorption of methanol from the activated carbon, although it resumed 100% removal quickly.

Finally, the mass loading was increased to about 5 g/m^3 packing/hr to test the column performance under varied conditions. The biofilter continued 100% removal during this phase. The control column performed similarly, except for a three-day period at the conclusion of the trial, when a slight reduction in performance was observed.

In conclusion, it appears that the column design, particularly use of packing material that not only provides adsorptive capacity, but also contributes to nutrient availability and moisture regulation in the column, is viable for supporting biological methanol removal. This packing material supported the growth of bacteria specifically enriched for methanol degradation and used as inoculum to the column as well as biological material of an unknown origin that was able to colonize the control column without specific inoculation. Comparisons of the abundance of microbial growth and bacterial diversity between the two columns and between different regions within each column in ongoing.

Abundance of bacteria colonizing the biofilter

After the conclusion of the feasibility trial, packing material was extracted sterilely and homogenized. Two grams of packing material was added to 10 mL of phosphate buffer and vortexed at 3,000 rpm in 15 second increments for two minutes. Supernatant from this mixture was serially diluted and spread plated on three types of agar plates (nutrient, nitrate mineral salts, and ammonium mineral salts). Nutrient plates contained a multi-carbon source, while both mineral salts plates were incubated under a methanol atmosphere. All plates were incubated at 30 deg. C for 72 hours, and then visible colonies were counted and averaged for four replicates of each plate type. Results for the inlet region of the biofilter are shown below.

Table 1. Enumeration of colony forming units from biofilter packing

	Column Inlet (CFU/g)
Nutrient	1.71E+09 (+/- 2.59E+07)
NMS	1.55E+09 (+/- 4.79E+07)
AMS	1.32E+09 (1.69E+08)

Bacterial Diversity of the Biofilter under Varied Operating Conditions

Throughout the operation of the biofilter and control column, packing material was removed from the inlet portion of the columns twice during each operating condition and analyzed for genetic diversity using optimized molecular techniques. At the conclusion of the trial, material was also collected from the three length-wise regions of the column. Two grams of packing material was added to 5 mL phosphate buffer and vortexed continuously at 3,000 rpm for two minutes. DNA was extracted from this mixture using MoBio UltraClean Microbial DNA extraction kits and amplified by polymerase chain reaction (PCR) using primers designed to

amplify a highly conserved region of the gene coding for methanol dehydrogenase, the enzyme responsible for methanol degradation in all known Gram negative methylotrophs. The PCR reaction mixture contains 1X $MgCl_2$-free PCR buffer, 1.5 mM $MgCl_2$, 100 M of each dNTP,1U *Taq* polymerase (all from Invitrogen), 0.5 M of each primer (Integrated DNA Technologies, Inc.), 2 L of template DNA, and sterile water to a final volume of 50 L. Reactions were conducted with an initial denaturation at 94 deg. C for 3 minutes, 30 subsequent cycles of 30 second denaturation at 94 deg. C, 30 second annealing using a touchdown program from 60-50 deg C for the first 20 cycles and 50 deg C for the final 10 cycles, and 45 second extension at 72 deg C.; with a final extension of five minutes at 72 deg. C. The PCR mixture and reaction program were optimized for specificity and yield. PCR products were checked on a 1.2% agarose gel and final concentrations estimated using ImageJ software (NIH) calibrated with a low DNA marker (50-2,000 bp, BioNexus).

Diversity of the bacteria colonizing the biofilter was assessed by analyzing PCR products using denaturing gel gradient electrophoresis (DGGE), a method in which PCR-amplified DNA is electrophoresed in a polyacrylamide gel with a linear gradient of denaturant to separate DNA fragments of the same length but slightly different sequences, as based on the relative electrophoretic mobility associated with the melting point of each sequence. DNA fragments were separated under DGGE conditions with a gel containing 6% acrylamide, 1X TAE buffer, a denaturing gradient of 35-65%, and an electrophoresis time of 5 hours at 150V using a DCode Universal Mutation Detection System Model 475 Gradient Delivery System (Bio-Rad). After staining with ethidium bromide and destaining, bands were visualized and photographed using a FisherBiotech Model 88A variable UV intensity Transilluminator and DCode DocIt software system (BioRad). Initial results for the first four DNA extraction periods for the biofilter and control column are shown below.

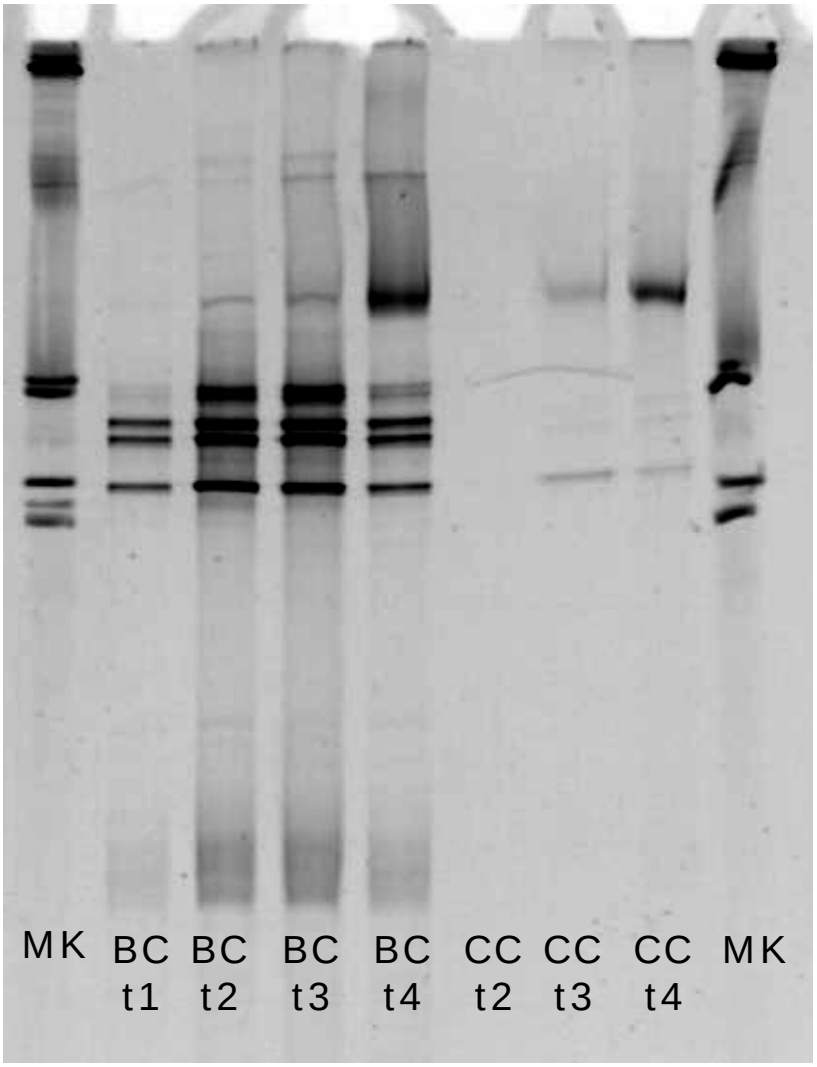

Figure 7. Bacterial diversity under varied operating conditions

As shown in Figure 7, each band on the gel potentially represents a single bacterial species in a mixture, and the intensity of each band represents the relative abundance of that species based on the DNA amplified by PCR. DNA present in amounts of 1% or less of the total mixture will likely not be shown. The PCR conditions used can bias the results, but given consistency in performing each reaction, the banding patterns can lend insight into the changing diversity in the columns. These results indicate that three primary species colonize the biofilter from inoculation and persist through each operating condition, although other species become active and dominating at lower methanol loading rates. It appears that two methylotrophic species could be responsible for colonizing the control column and effecting methanol degradation there. It is possible that this contamination occurred within the system, from one column to the other, rather than from ambient exposure. This analysis is being repeated for additional time periods

during column operation and at the end of the column, as well as for the different regions within each column. Additionally, diversity will be compared with the original inoculum as added to the biofilter and as maintained in a batch culture. Bands have also been excised from the gel, purified, and reamplified in preparation for DNA sequencing of the most abundant species under each condition. These comparisons should add insight into the ecology of the biofilter as it relates to performance under varied operating conditions.

Future Studies

Next quarter, methanol removal will be tested using the second generation pilot-scale reactor packed with silica-titania coated chemical packing material. In addition, LCA data collection and analysis will continue next quarter.

Patents: None filed

Publications/Presentations:

Mazyck, D.W., Wu, C.Y., Lindner, AS., Sheahan, R., and Jain, A. *TiO_2-coated carbon for the removal of VOCs*. TAPPI Paper Summit, 2004.

Stokke J.M., Mazyck D.W., Wu C.Y. *Comparison of titania-doped sorbents for VOC/HAP control.* Air and Waste Management 98th Annual Conference and Exhibition. Minneapolis, MN. June 21-24, 2005.

Stokke J.M., Mazyck D.W., Wu C.Y., Sheahan, R. *Development of Silica-Titania Composites in a Packed-Bed Photocatalytic Reactor for the Removal of VOCs and HAPs.* Environmental Progress. Submitted March 2006.

Tao, Y., Wu, C.Y., Mazyck, D.W. *Development of a TiO_2/Activated Carbon Composite Photocatalyst by Pore Volume Impregnation for Treatment of VOCs and HAPs.* Air & Waste Management Association. Minneapolis, MN on June 21-24, 2005.

Tao, Y., Wu, C.Y., Mazyck, D.W. *Development of a TiO2/AC composite photocatalyst by dry impregnation for the treatment of methanol in humid airstreams*. Industrial & Engineering Chemistry Research 44 (19), 7366-7372, 2005.

Poster Presentations:

Babbitt, C. Lindner, A.S. *A biological activated carbon reactor for reducing emissions of hazardous air pollutants from pulp and paper mills*. Air and Waste Management 98th Annual Conference and Exhibition. Minneapolis, MN. June 21-24, 2005.

Mazyck, D., Lindner, A., Wu, C., Stokke, J. An Innovative Titania-Activated Carbon System for the removal of VOCs and HAPs from Pulp, Paper, Paperboard Mills, and Wood Products

Facilities with In-situ Regeneration Capabilities. ITP Forest Products Peer Review. Atlanta, GA. April 2006.

Tao, Y., Wu C.Y., Schwartz, S., Mazyck, D.W. *Development of a TiO2/Activated Carbon Composite Photocatalyst by Pore Volume Impregnation for Treatment of VOCs and HAPs.* Air and Waste Management Conference. Orlando, FL. October 27, 2004.

Milestone Status Table:

ID Number	Task / Milestone Description	Planned Completion	Comments
1	Development of Materials		
1.1	Literature Review	8/31/03	Review journals and patents
1.2	Assess Commercial Products	9/30/03	Characterize pros and cons
1.3	Tailor Products with Wood Biomass	11/31/03	Optimization for application
1.4	Assess silica as a sorbent	9/30/03	Characterize pros and cons
1.4	Optimize Coating Strategy	1/31/04	Focus on sol gel
1.5	Quantify Regeneration Efficiency	3/31/05	Performance testing
2	Pilot Design and Testing	9/30/03	
2.1	Site Visits	10/31/03	Mill assessments
2.2	Design System	12/31/03	Schematics/Bill of Materials
2.3	Construct and Evaluate Bench Apparatus	3/31/04	For prototype research
2.4	Construct Pilot Plant	12/31/04	For field site installation
2.5	Evaluate Pilot Plant	3/31/05	Shakedown tests
3	Field Tests	2/28/06	
3.1	Data Collection	2/28/06	Performance characterization
3.2	Assess bio/silica/carbon synergies	11/31/05	
3.3	Modify Pilot Plant	12/31/05	If necessary
4	Life Cycle Analyses	3/31/06	
4.1	Environmental	3/31/06	
4.2	Energy	3/31/06	
4.3	Economic	3/31/06	

Approved Budget Data:

Phase / Budget Period			DOE Amount	Cost Share	Total
	From	To			
Year 1	4/03	3/04	434,629	156,643	591,272
Year 2	4/04	3/05	650,617	167,574	818,191
Year 3	4/05	3/06	553,503	143,744	697,247
Year 4					
Year 5					
Totals			1,638,749	467,961	2,106,710

Second Project Year Spending Plan:

	YEAR 2				
	Feb-Mar 2004	2ndQ 2004	3rdQ 2004	4thQ 2004	TOTAL 2004
UF Personnel					
Faculty	3,440	9,855	6,375	4,170	23,840
Graduate students	12,180	34,900	22,585	14,770	84,435
Fringe	900	2,580	1,670	1,096	6,246
TOTAL LABOR & FRINGE	16,520	47,335	30,630	20,036	114,521
Supplies	1,645	4,710	3,045	1,990	11,390
Travel	1,970	5,650	3,655	2,390	13,665
NCASI	19,715	56,475	36,545	23,900	136,635
MSI	54,520	156,200	101,075	66,100	377,895
Other direct costs (publications, analysis, tuition)	4,180	11,980	7,755	5,070	28,895
TOTAL DIRECT COSTS	98,550	282,350	182,705	119,486	683,001
INDIRECT COSTS	9,653	27,656	17,896	11,704	66,909
TOTAL DIRECT & INDIRECT	108,203	310,006	200,601	131,190	750,000

Index of Award CID Numbers

Index of Award CID Numbers